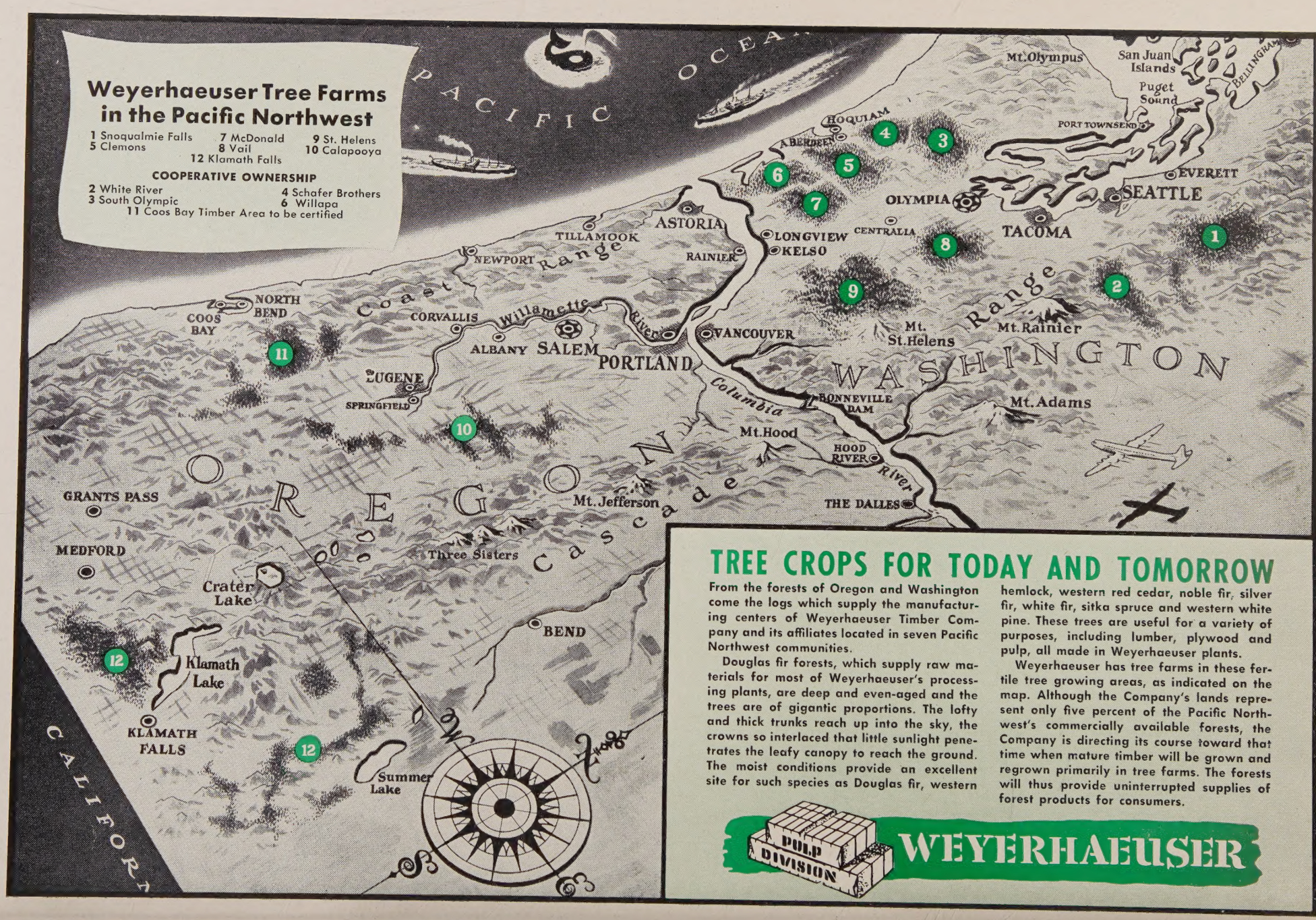
# Tappi

PUBLISHED BY THE TECHNICAL ASSOCIATION OF THE PULP AND PAPER INDUSTRY

FEBRUARY 1950




## TREE CROPS FOR TODAY AND TOMORROW

From the forests of Oregon and Washington come the logs which supply the manufacturing centers of Weyerhaeuser Timber Company and its affiliates located in seven Pacific Northwest communities.

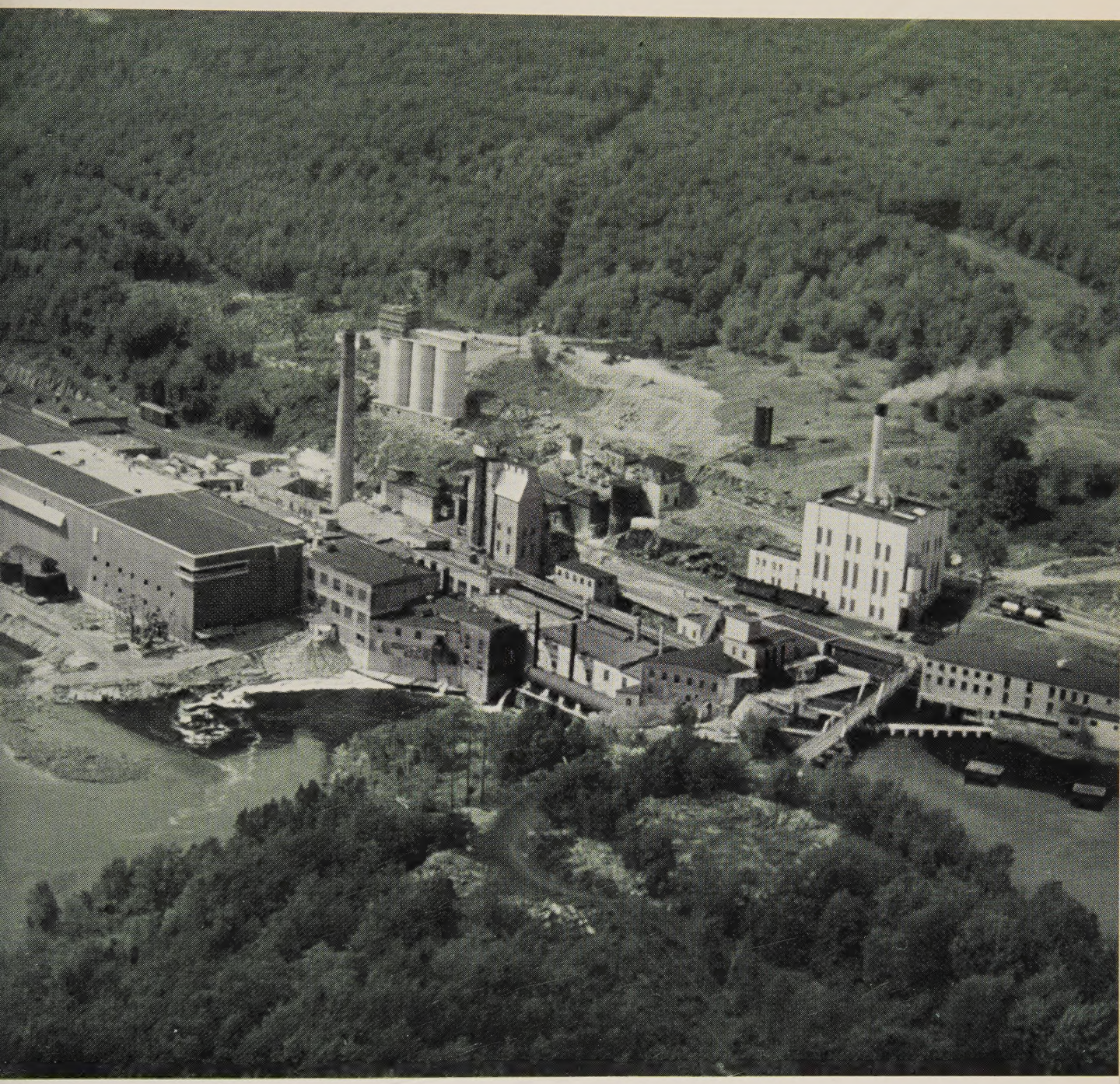
Douglas fir forests, which supply raw materials for most of Weyerhaeuser's processing plants, are deep and even-aged and the trees are of gigantic proportions. The lofty and thick trunks reach up into the sky, the crowns so interlaced that little sunlight penetrates the leafy canopy to reach the ground. The moist conditions provide an excellent site for such species as Douglas fir, western hemlock, western red cedar, noble fir, silver fir, white fir, sitka spruce and western white pine. These trees are useful for a variety of purposes, including lumber, plywood and pulp, all made in Weyerhaeuser plants.

Weyerhaeuser has tree farms in these fertile tree growing areas, as indicated on the map. Although the Company's lands represent only five percent of the Pacific Northwest's commercially available forests, the Company is directing its course toward that time when mature timber will be grown and regrown primarily in tree farms. The forests will thus provide uninterrupted supplies of forest products for consumers.




WEYERHAEUSER

# Kimberly-Clark: magazine and book



In this modern plant at Niagara, Wisconsin, a substantial part of Kimberly-Clark Corporation's high-quality book and magazine paper is being produced. It is the northernmost mill operated by K-C in the United States. Machinery and equipment have been kept up-to-date, the latest step being the addition, in 1948, of a 246″ High Speed Fourdrinier Machine and Super Calender, both by Beloit . . . builders of fine paper-making machinery for industry leaders since 1858.—*Beloit Iron Works, Beloit, Wisconsin.*

*WHEN YOU BUY BELOIT . . . YOU BUY MORE THAN A MACHINE!*

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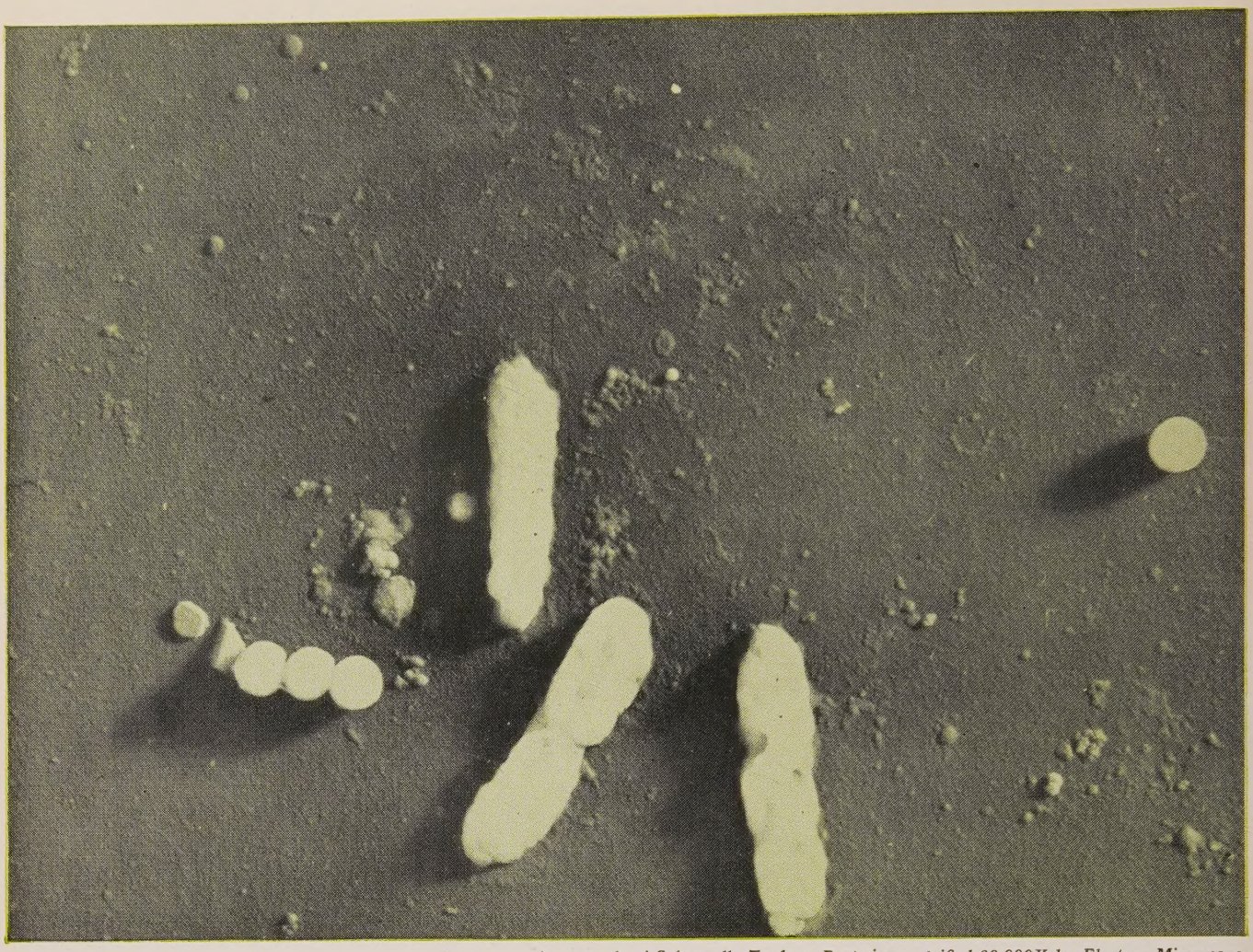

*An example of Salmonella Typhosa Bacteria, magnified 30,000X by Electron Microscope.*

# Are Bacteria Biting Into Your Profits?

During many production stages from the raw stock to the finished product, bacteria and mold are an ever present threat to the paper industry. These microorganisms cause mildew on lap stock, "sour" pulp slush, and attack glues, making them lose adhesive strength.

Such attacks cost time and money and result in shrinking profits. Like many leading paper mills, you can prevent these costly wastes with Dowicides. Dowicides, Dow's industrial germicides and fungicides, provide proper microbial and mold control. They will help eliminate souring and spoilage.

Don't let bacteria and mold bite into your profits. Investigate Dowicides today. Dow maintains extensive laboratory facilities to help you with your problem. Contact your nearest sales office or write direct.

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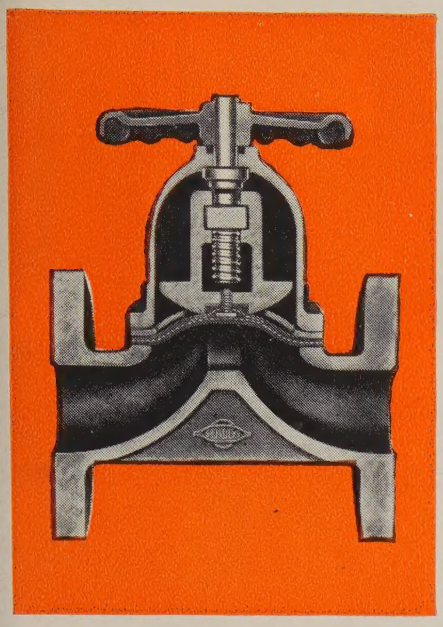
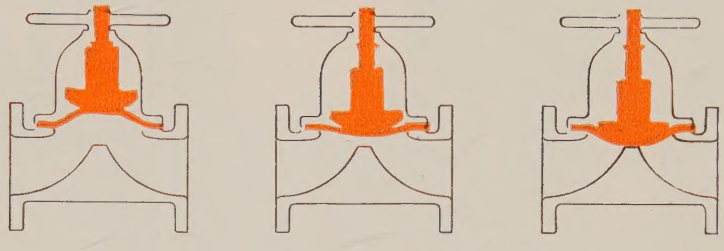
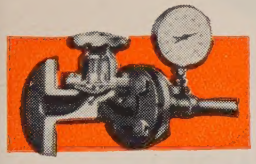


# If you need a diaphragm valve you need all the advantages of diaphragm closing



## Check these popular features of

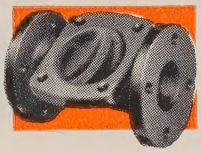
# GRINNELL-SAUNDERS DIAPHRAGM VALVES



**Diaphragm gives leak-tight closure against grit, scale, solid matter . . .** Even when something as substantial as an 8-penny nail is trapped on the weir of a rubber lined valve, as illustrated, the resilient diaphragm, plus the large area of contact, gives leak-tight closure against pressure or vacuum.

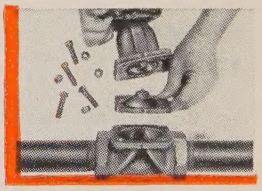
**Diaphragm lifts high for streamlined flow in either direction . . .** Smooth, streamlined passage, without pockets, prevents trapping of sludge and reduces frictional resistance to a minimum—irrespective of direction of fluid flow. No disc holder in fluid stream. Grinnell-Saunders Diaphragm Valves are self-draining when installed with the spindle at 15 degrees above the horizontal position.

**Diaphragm absolutely isolates working parts from fluid . . .** There's no "if" about the way a continuous, one-piece diaphragm seals off the working parts from fluids; no perforation or puncture in the diaphragm where fluid or gas can possibly leak by the valve spindle. No sticking, clogging or corroding of working parts. Valve lubricant cannot contaminate the fluid in the line.



**Diaphragm, body and lining materials to meet particular conditions . . .** Bodies stocked in cast iron, malleable iron, stainless steel, bronze and aluminum: other materials on special orders. Valve bodies lined with lead, glass, natural rubber or neoprene. Diaphragm materials of natural rubber or synthetics. Working pressure, 150 lbs.; maximum temperature, 180 °F.

**Diaphragm is only part that normally wears and needs replacement . . .** Depending on the type of service, it may last for years, particularly since the compressor and finger plate combine to support the diaphragm in all positions. The diaphragm can be replaced quickly without removing valve from line. No refacing or reseating. No disc holder to require tightening or replacing. No packing glands to demand attention.



*Write today for the new Grinnell-Saunders Diaphragm Valve Catalog 4-S.*

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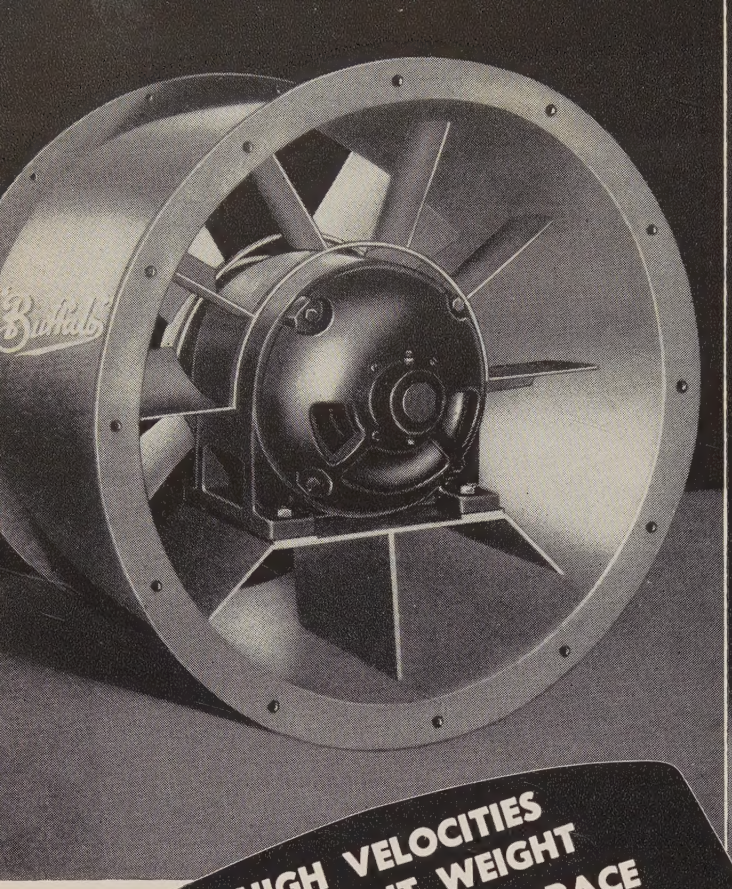

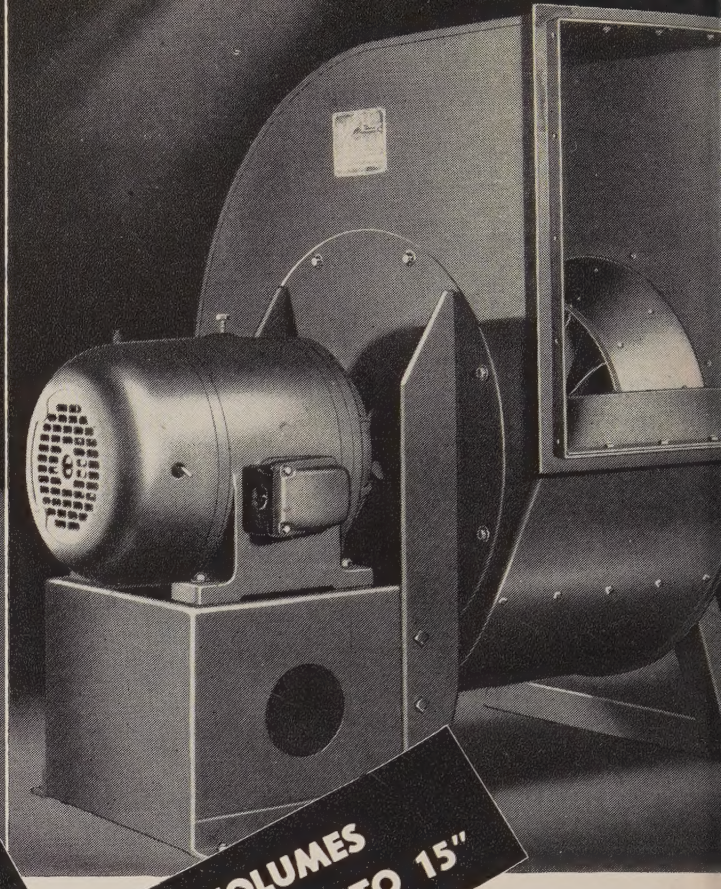

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# "Buffalo" GIVES YOU YOUR CHOICE FOR BEST RESULTS ON THE JOB!

"Buffalo" builds both Axial Flow and Centrifugal Fans because ONE type alone can't give EVERY characteristic without sacrificing efficiency. "Buffalo" Axial Flows give you the compact, lightweight design so desirable in those "cramped quarters" jobs. Their efficiencies are very high where static pressures are not much above 1½" and duct connections are straight. "Buffalo" does build axials for high pressures on special installations—where space is the major consideration. WRITE FOR BULLETIN 3533-C for complete facts and comparative performance curves.

Generally, if you want to move large volumes of air, the "Buffalo" Limit-Load Fan is your logical choice. It will give you good efficiencies on systems of fairly high pressure, and even where installed near duct curves. Like the "Buffalo" Axial Flow Fan, it is non-overloading so that motor size may be picked close to capacity rating—and all wheels are dynamically balanced for exceptionally smooth, quiet delivery. Closely graduated sizes give you a range of choice from 600 cfm to over 500,000 cfm. WRITE FOR BULLETIN 3675.

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FEBRUARY 1950

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# Stream-Flow Vat System

Flow Spreader for Cylinder Machines

# —a story of product improvement

It was just a few years ago that a box with a cylinder mold was considered a vat . . . and what this vat couldn't accomplish was supposedly beyond the realm of Cylinder Machines.

Then came the Goldsmith Stream-Flow Vat System—an individual forming machine designed and built by Puseyjones with the same thought and care as Puseyjones Fourdriniers. Step by step the Stream-Flow Vat System has been improved. Here are some of the outstanding features—standard, but optional—of the latest Stream-Flow design:

(1) Vacuum Cylinder Mold . . . One of the most important improvements in the history of Cylinder Machines. Formation is greatly improved due to use of vacuum and elimination of spoke disturbance. For single cylinder or multi-cylinder machines.

(2) Stream Flow Vat . . . Progressive improvements and simplifications have been made, including use of stainless steel instead of bronze for flexible, adjustable vat circle; independent front, bottom and back circle adjustments reading directly on dials; an ingenious toggle-operated bleeding and dumping arrangement.

(3) Flow Spreader . . . New compact design. Hydraulic nozzle "squeezes" the flow by decreasing area and increasing velocity during spreading.

(4) Up-Flow Head Box . . . New design assures steady feeding of the vat and permits air to escape.

For better formation, higher speeds, a wider range of stock freenesses proven in operation on a wide variety of grades—from thin cylinder papers to roofing felt — investigate the Stream-Flow Vat System. Talk to Puseyjones engineers about doing the job in several steps — the proof of performance is in the operation. Write us today.

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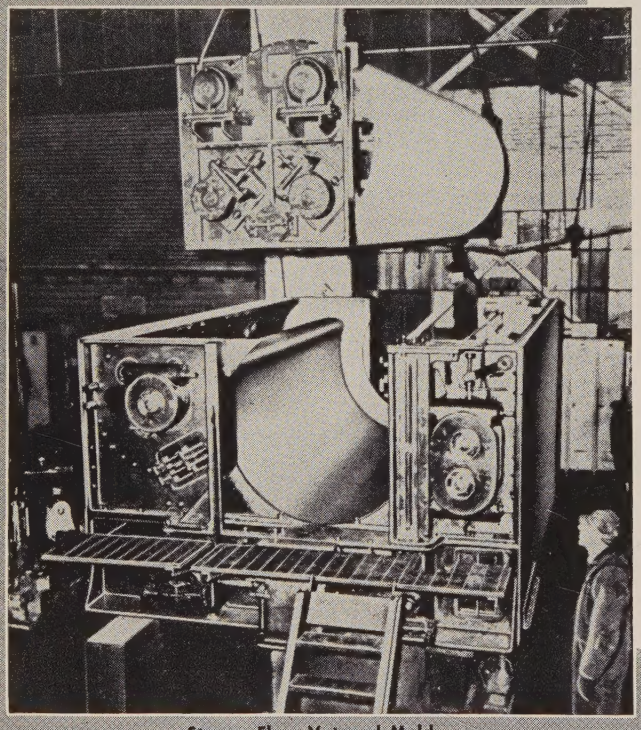
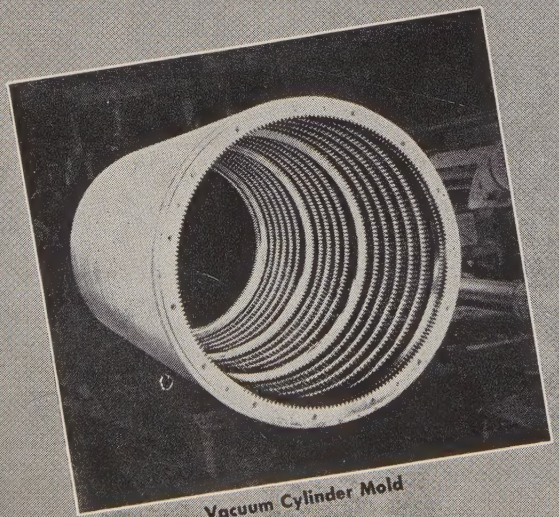
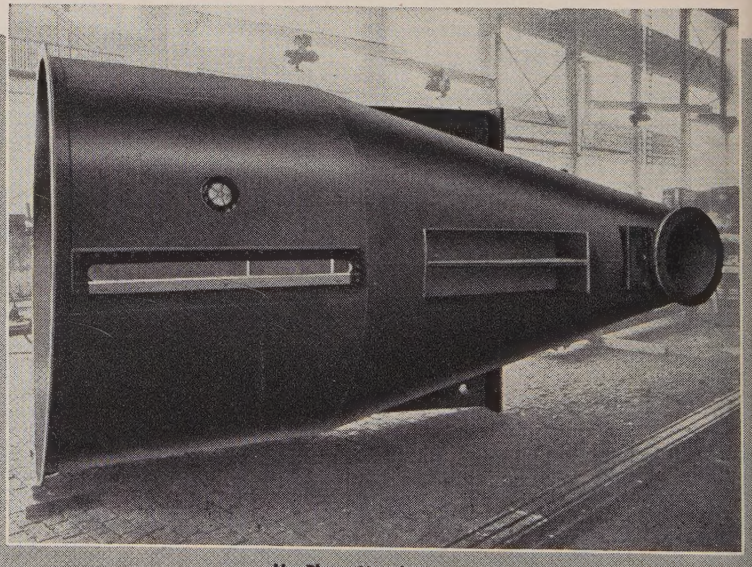


Stream Flow Vat and Mold



Vacuum Cylinder Mold



Up-Flow Head Box

# NOPCO KFS* CUTS FOAM ALL ALONG THE LINE

Wherever foaming occurs—on screens, in rifflers and washers, and on paper machines—you'll find Nopco's newly-developed KFS an outstanding foam killer. It practically eliminates obstinate foam conditions within a *matter of minutes.*

If results with defoamers you have already used have not proved satisfactory, it is very probable that KFS is the one you are seeking. For this new foam destroyer is stable to chemicals such as alum and chlorine, and to hard water. It can do a real job *all along the line,* no matter how great the volume or speed of production.

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Added to digester effluent, KFS cuts foam to negligible proportions.

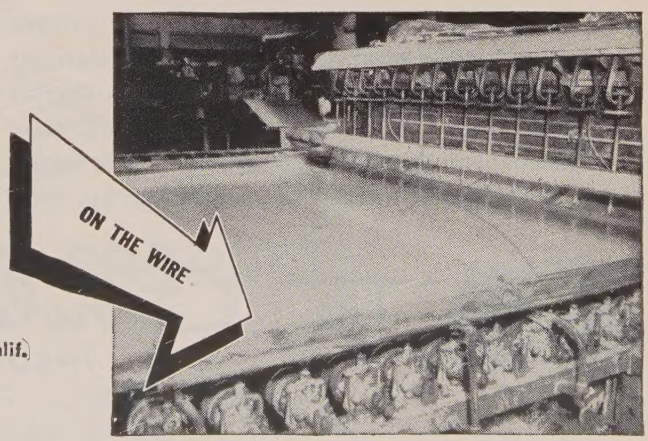
Added to bleach plant effluent, which contains chlorine-lignin combinations and foams heavily, KFS does an exceptional job of foam control—making it easy to dispose of waste liquors.

Added to stock immediately before it passes through screens, rifflers and washers, KFS prevents foam formation during processing.

## IN THE PAPER MILL

When added to stock just preceding any point where foaming occurs, KFS dissipates foam effectively. Thus foaming can be eliminated on the screen, in the vat, or on the wire. The results are: better fibre dispersal, reduced breaks, *improved sheet formation.* In addition, higher machine speeds are made possible *without sacrifice of quality.*

Nopco KFS is easily made into a stable emulsion, by agitation with a paddle. Order enough for a trial run—and see for yourself how advantageous this new defoamer can be.

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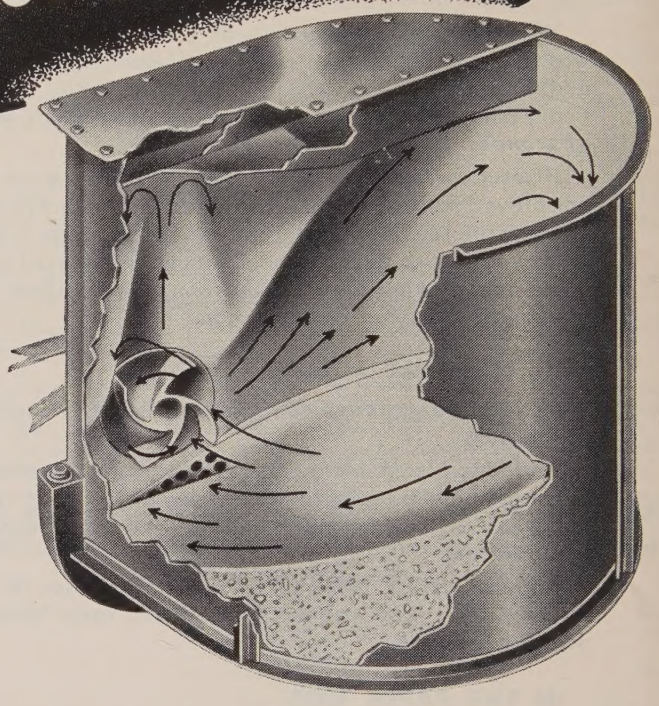

## May Be Used Continuously or for Batch Pulping of Clean Stock

At last, a pulper that gives you the features you've always wanted. Truly revolutionary in its application of tried and proved pulping principles, the Cowles Pulper achieves efficiency and economy unsurpassed by anything else on the market.

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The side-drive design of the machine permits circulation of high consistencies with relatively small high-speed impellers . . . hence, sharply reduces the cost of the impeller and drive mechanism. A steeply-sloped bottom facilitates rapid drainage and will handle batches from ⅓-size to full-size with equal efficiency. There is virtually no splash at any operating level. Submergence is excellent at all levels and consistencies.

Like all Cowles equipment, Cowles Pulpers are soundly engineered and ruggedly constructed for long, trouble-free service. They embody proved hydraulic principles that yield high output with low power consumption. Write or wire for complete details.

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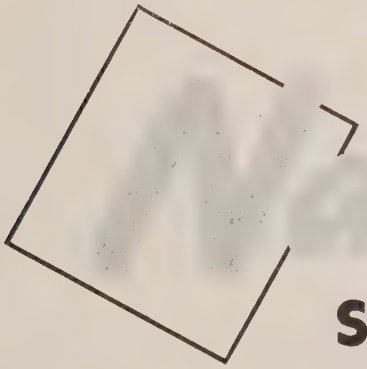

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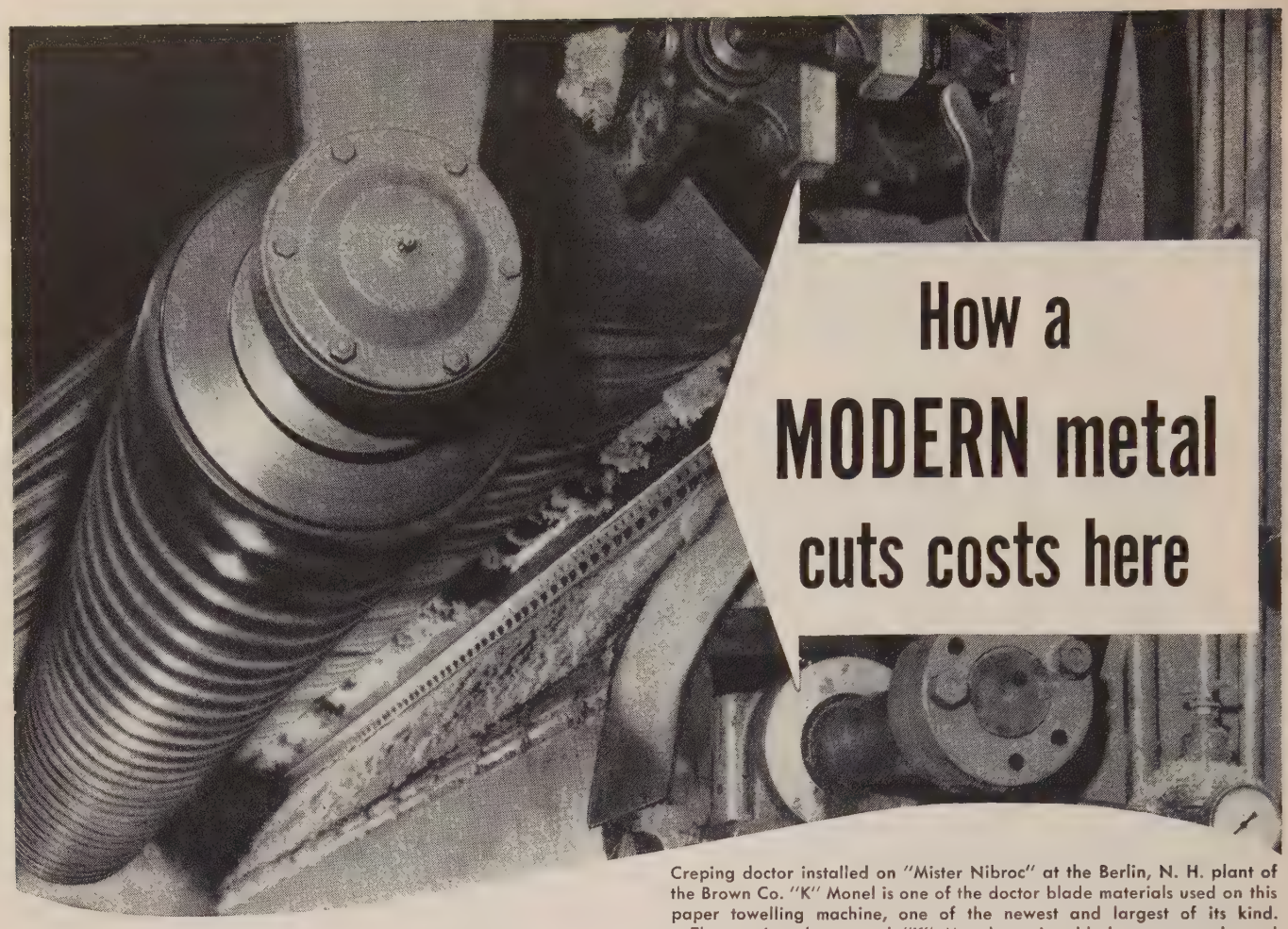

# How a MODERN metal cuts costs here

Creping doctor installed on "Mister Nibroc" at the Berlin, N. H. plant of the Brown Co. "K" Monel is one of the doctor blade materials used on this paper towelling machine, one of the newest and largest of its kind. The creping doctor and "K" Monel creping blades were made and furnished by LODDING ENGINEERING CORP., Worcester, Mass.

Wear and corrosion...worst enemies of your doctor blades . . . *can* be controlled.

The secret of long-lasting, low-maintenance blades lies in using a metal that fits your operating conditions.

"K"* Monel is just such a metal. Consider its unique combination of desirable properties:

- **Non-rusting, highly corrosion-resistant.**
- **Strength and hardness greater than structural steel.**
- **Heat-treatable for maximum properties.**
- **Easy to machine; takes a high polish.**

So much for theory. Now . . . what about practice?

Many of the nation's busiest board and paper mills report outstanding service from "K" Monel doctor blades, in both dry and wet end applications.

"K" Monel blades, operating on modern high-speed paper machines, have given up to *18 times* longer service than blades of other commonly used metals. Less honing and less regrinding are required. Corrosion ceases to be a threat to blade life. Maintenance costs drop sharply.

Monel* has long been a preferred metal for many other paper mill applications, too . . . screens, save-alls, jordan bars, winding wire, head boxes, rolls and roll covers.

Why not investigate the money-saving possibilities of the versatile Inco Nickel alloys for your own Mill? A note to Inco will bring you full information. Write today!

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# Paper Product Pointers
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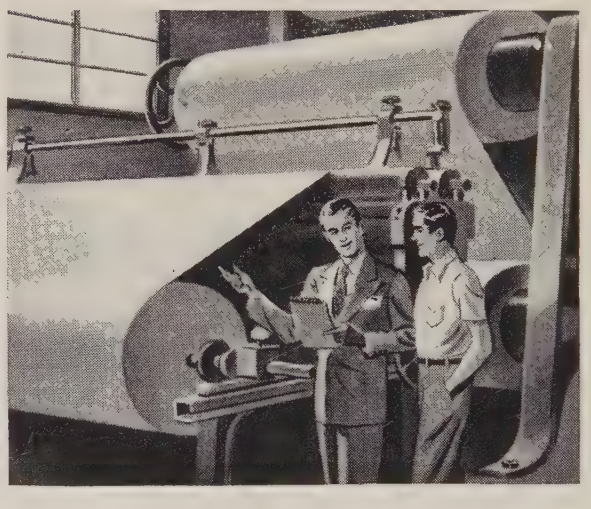
Our Technical Service Staff is available to help manufacturers develop new papers through the proper application of Cyanamid chemical products. Present papers may be improved quality-wise and processes made more efficient through their experienced counsel.

If you have a "paper" problem, the services of the Technical Service Staff are at your call with no obligation. The specialized skill and experience of these paper chemical specialists, backed by our Stamford Research Laboratories, are as essential a component of our products as are the quality and uniformity inherent in every batch of Cyanamid paper chemicals.



Look us up at the TAPPI Meeting in New York in February. We will be glad to discuss your paper chemical problems with you.

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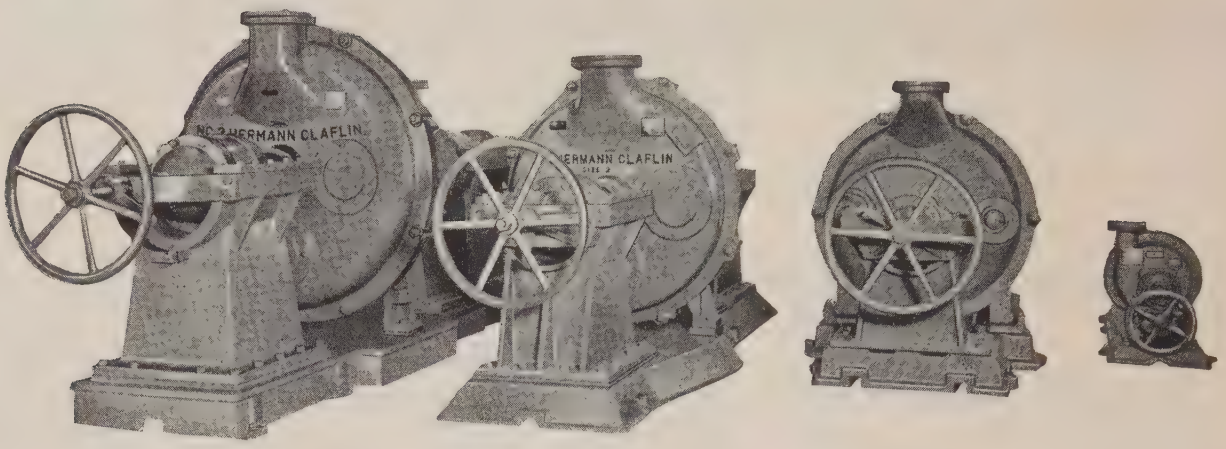


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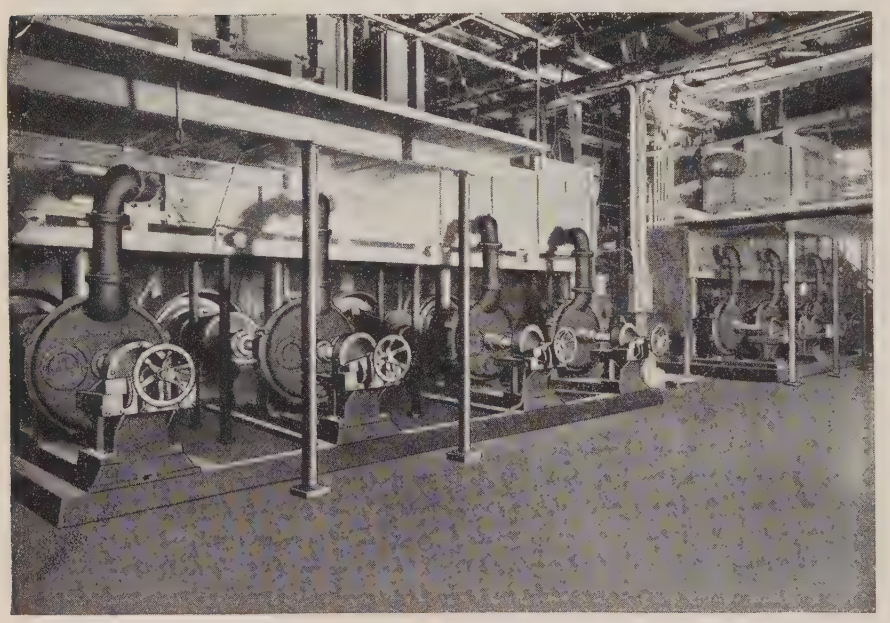

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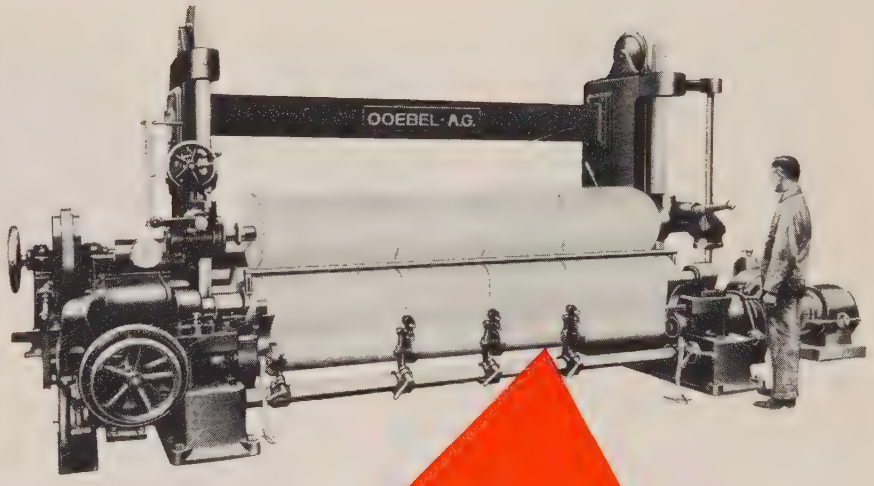


# TAPPI Annual Meeting

## *Commodore Hotel, New York, N. Y., February 20–23, 1950*

### MONDAY, FEB. 20, 1950

***10:00 a.m. General Session (East Ball Room)***

A. E. Bachmann, Missisquoi Corp., Sheldon Springs, Vt., *presiding*

1. Secretary's Report, by R. G. Macdonald.
2. President's Address, by A. E. Bachmann.
3. Tellers Report.
4. Joint Textbook Committee Report, by R. S. Kellogg.
5. "The Use of Radioisotopes for Process Control in the Pulp and Paper Industry," by A. P. Schreiber, Tracerlab, Inc.

***10:00 a.m. Wax Testing (Room B-C)***

A. M. Heald, Marathon Corp., Menasha, Wis., *presiding* Meeting of TAPPI-ASTM Joint Committee on Petroleum Wax.

***2:00 p.m. Committee Meetings (Grand Ball Room)***

All committees of the Association will hold separate meetings to discuss committee projects and future plans.

### TUESDAY, FEB. 21, 1950

***9:15 a.m. Papermaking (Cylinder Machine) (Grand Ball Room)***

J. J. Harrison, Michigan Carton Co., Battle Creek, Mich., *presiding*

1. "Vortraps as Applied to Paperboard Manufacture," by H. E. Ingalls, Nichols Engineering and Research Corp.
2. "Application of the Duotrol to Jordans and Refiners," by T. Agronin, Shartle Bros. Machine Co.
3. "Rapid Length Classification of Pulp," by Arthur E. Reed, Gaylord Container Corp. and J. d'A. Clark.
4. "Development of the Cowles Pulper," by J. M. Kelly, Kelly-Forsyth Corp., Ithaca, N. Y. and Edwin Cowles, The Cowles Co., Cayuga, N. Y.
5. "The Dominion Engineering Vacuum Vat," by C. S. V. Hawkings, Price Brothers & Co., Ltd.

***9:15 a.m. Fibrous Agricultural Residues (East Ball Room)***

S. I. Aronovsky, Northern Regional Research Laboratory, Peoria, Ill., *presiding*

1. "Developments in the Methods of Treating Strawboard Waste," by D. E. Bloodgood, Purdue University.
2. "Improved Straw Pulp for Corrugating Paper," by H. M. Sutcliffe, and S. I. Aronovsky, Northern Regional Research Laboratory, R. M. Wilkinson, Central Fibre Products Co., W. A. Phillips, and E. R. Carpenter, Terre Haute Paper Co.
3. "Processes Being Used in Europe for the Manufacture of Straw Pulp," by Joseph E. Atchison, Economic Cooperation Administration.
4. "A Laboratory Corrugator for 9-Point Medium," by Norval F. Wilson, Alton Box Board Co.

***9:15 a.m. Wet Strength (West Ball Room)***

K. W. Britt, Scott Paper Co., Chester, Pa., *presiding*

1. "Mechanism of Retention of Beater Added Urea Resins," by M. N. Fineman and I. J. Gruntfest, Rohm & Haas Co.
2. "Neoprene Latex in the Paper Industry," by R. H. Walsh. W. W. Pockman, J. R. Gallaway, E. P. Hartsfield, and H. H. Abernathy, E. I. DuPont de Nemours & Co., Inc.
3. "Permanence of Wet Strength Paper," by C. S. Maxwell, and W. F. Reynolds, American Cyanamid Co.
4. "The Instron Tensile Tester," by George S. Burr and Harold Hindman, Instron Engineering Corp.
5. "The Softening Mechanism of Paper Plasticizers," by P. E. Nethercut, Watervliet Paper Co. (By Title).

***2:00 p.m. Chemical Engineering (Room B-C)***

J. R. Lientz, Union Bag & Paper Corp., Savannah, Ga., *presiding*

1. "The Role of the Chemical Engineering in the Pulp and Paper Industry," by G. W. E. Nicholson, Union Bag & Paper Corp.
2. "Chemical Engineering Unit Operations in the Pulp and Paper Industry," by W. L. Badger, Consulting Engineer.
3. "Stainless Clad Steel Equipment for Digesters," by W. H. Funk and E. H. Wyche, Lukens Steel Co.
4. "Corrosion in Sulphate Pulp Digesters," by T. C. Johnson, Union Bag & Paper Corp.
5. "The Stacomizer—A Continuous Roller Press," by R. J. Chambers and K. C. Logan, Northeastern Paper Products, Ltd.

***2:00 p.m. Water (Grand Ball Room)***

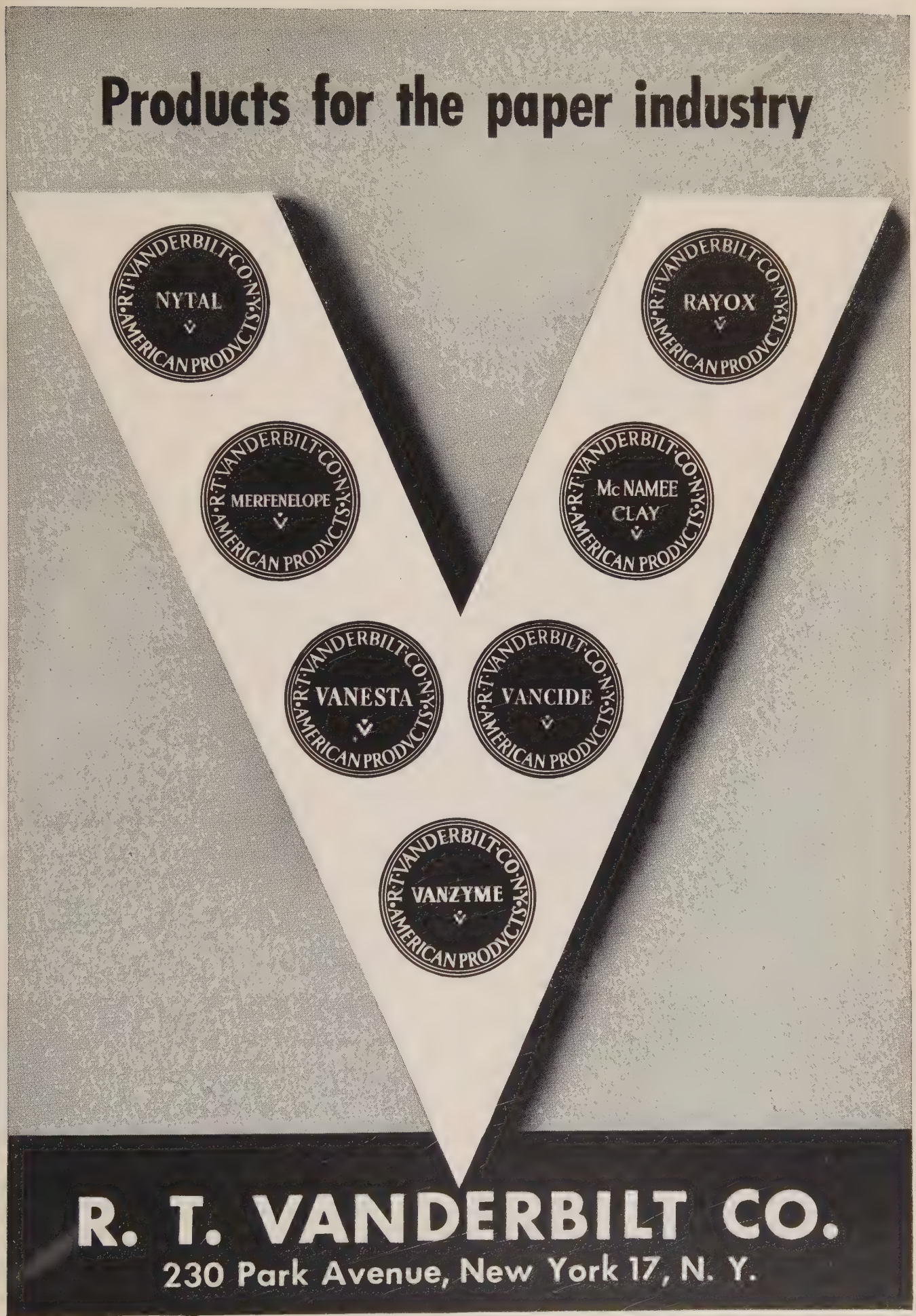
A. S. Erspamer, P. H. Glatfelter Co., Spring Grove, Pa., *presiding*

1. "Industry's Stake in the Ohio River Pollution Abatement Program," by Edward J. Cleary, Ohio River Valley Sanitation Commission.
2. Paper by Harry Gehm, National Council for Stream Improvement.
3. "The Waco Filter," by Joseph K. Perkins, Improved Paper Machinery Co.
4. "White Water Treatment," by F. A. Eidsness, Infilco, Inc.
5. "Economic Considerations in White Water Treatment," by V. J. Calise, Graver Water Conditioning Co.
6. "Control of Sewer Losses," by John G. Dobson, Foxboro Co. (By Title).
7. "Some Factors Affecting Slime Formation and Freeness of Board Mill Stock," by Willem Rudolfs and Nelson L. Nemerow, Rutgers University (By Title).

***2:00 p.m. Plastics (West Ball Room)***

E. C. Jahn, N. Y. State College of Forestry, Syracuse, N. Y., and C. J. Straka, Westinghouse Electric Corp, Trafford, Pa., *presiding*

1. "Preparation and Properties of Various Papers for Lamination," by C. V. Baird, Forest Products Laboratory.
2. "The Effect of Paper Type and Structure on the Physical Properties of Phenolic Resin Bonded Laminates," by M. P. Seidel, Westinghouse Electric Corp.
3. "The Effects of Paper Type and Structure on the Moisture Dimensional Stability of Phenolic Resin Bonded Laminates," by M. P. Seidel.
4. "The Beater Addition Process for Producing Laminated Papers," by F. H. Snyder, L. Spiwak, and A. E. Moyer, Snyder Chemical Corp.
5. "Formulation and Properties of Laminating Varnishes," by C. B. Bennett, Catalin Corp. of America.
6. "Investigation of the Loss Angle and Dielectric Constant of Cellulose," by Charles R. Calkins, Institute of Paper Chemistry (By Title).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*2:00 p.m. Alkaline Pulping (East Ball Room)*

HENRY VRANIAN, Chesapeake Corp., West Point, Va., *presiding*

1. "The Pulping of Scrub Oak (Quercus Laevis) by the Kraft Process," by R. L. Harvin, C. W. Rothrock, and W. J. Nolan, University of Florida and G. B. Hills, Jr., St. Joe Paper Co.
2. "Chemical Cleaning of Black Liquor Evaporators," by H. H. Walther, Dowell, Inc.
3. "The Operation of the Multiroll Beater-Refiner in a Kraft Mill," by Milton R. Roberts, Hudson Pulp and Paper Corp.
4. "The Effect of Roll Load in the Beating of Southern Kraft Pulp," by C. S. Walseth and C. J. Geijer, Union Bag & Paper Corp.
5. "The Effect of Kraft Pulp Mill Wastes on Fish Life," by W. B. Van Horn, Institute of Paper Chemistry.
6. "The Synthesis of Terpene Carboxylic Esters from Crude Sulphate Turpentine," by Torsten Hasselstrom, C. E. Balmer, N. E. Kennedy, and H. W. Coles, Philadelphia Quartermaster Depot.

## WEDNESDAY, FEB. 22, 1950

*9:15 a.m. Water (East Ball Room)*

C. M. CONNOR, W. C. Hamiltor & Louis, Inc., Miquon, Pa., *presiding*

1. "Ground Waters," by William F. Guyton, U. S. Geological Survey.
2. "Process Water and White Water Filtration with the Automatic Backwash Sand Filter," by F. C. Schad, Hardinge Co., Inc.
3. "Corrosion Control in Water Treatment," by W. Tanzola, W. H. and L. D. Betz.
4. "Electrolytic Coagulation," by Paul G. Stephan, Alhydro, Inc.
5. "Flow Instrumentation in the Pulp and Paper Industry," by G. Douglas Zimmerman, Fischer & Porter Co. (Canada) Ltd.

*9:15 a.m. Coating (Grand Ball Room)*

J. H. HEUER, St. Regis Paper Co., Deferiet, N. Y., *presiding*

1. "Starch and Its Application in Machine Coating," by Ralph W. Kerr, Corn Products Refining Co.
2. "Enzyme Conversion and Its Application to Machine Coatings," by D. A. Hughes and W. L. Craig, R. T. Vanderbilt Co.

*Note:* A motion picture film—Effect of Amylase on Starch—will be shown by Sutton Redfern, Standard Brands, Inc.

3. "A Method of Preparing Water-Resistant Starch Coatings by the Use of Antimony Compounds," by Lawrence Gussman, N. M. Frisch, and F. J. Sweeney, Stein, Hall & Co.
4. "The Rheology of Paper Coatings," by William R. Willets, Titanium Pigment Corp.
5. "Casein Adhesive in Roll Coating," by J. W. Smith, R. T. Trelfa, and H. O. Ware, Hercules Powder Co.
6. "The Reverse Roll of Principle in Coating," by Lawrence W. Egan, Frank W. Egan & Co.

*Note:* Papers not completed at this session will be presented at a Joint Coating-Graphic Arts Session in the afternoon.

*9:15 a.m. Microbiological (West Ball Room)*

R. F. DELONG, Marathon Corp., Rothschild, Wis., *presiding*

1. "The Preparation and Testing of Mold Resistant Wrapping Papers," by Louis C. Barail, U. S. Testing Co.
2. "Performance of Paper Machine Wet Felts. I. Preliminary of Microscopic Observations of the Effect of Microorganisms on the Plugging of Felts," by Stanley Buckman, Buckman Laboratories.
3. "Some Further Observations on *Penicilium Roqueforti Thom*," by John W. Appling, S. J. Buckman, and Conan D. Cash, Buckman Laboratories.
4. "Further Studies on the Lethal Effect of Drier Rolls upon Bacteria in Paperboard, by B. F. Shema, Institute of Paper Chemistry.
5. "Slime Acclimatization," by Paul A. Sartoretto, W. A. Cleary Corp.
6. "Slime Measurement," by John H. Bardsley, St. Lawrence Paper Mills and L. A. McMeown, Consultant.

*2:00 p.m. Coating and Graphic Arts (Grand Ball Room)*

R. C. SIMMONS, Government Printing Office, Washington, D. C., and J. H. HEUER, *presiding*

1. "The Picking of Paper and Its Relation to Ink Tack," by Andries Voet and Claude F. Geffken, J. M. Huber Corp.
2. "New Inks for the Graphic Arts," by George Cramer, Sinclair & Valentine Co.
3. "The Evaluation of Coated Paper Surfaces for Printing Ink Absorption," by Andries Voet and John S. Brand, J. M. Huber Corp.
4. "Safety Papers," by Francis L. Simons, Crane & Co. (By Title).

*2:00 p.m. Papermaking (Fourdrinier) (East Ball Room)*

NOEL R. PHILLIPS, Champion Paper & Fibre Co., Canton, N. C., *presiding*

1. "A Stain for Use in the Microscopy of Beaten Fibers," by Francis L. Simons, Crane & Co.

*Symposium on Paper Machine Slices*

2. (a) "Slices for Rag Content Papers," by Robert W. Pattison, Cellulose Fibre, Inc.
   (b) "Slices for Lightweight Papers," by F. O. Boylon, Crown Zellerbach Corp.
   (c) "Slices for Newsprint," by Vernon P. Tipka, News Print Service Bureau.
   (d) "The Ideal Slice," by S. A. Staege, Black-Clawson Co.

*2:00 p.m. Containers (West Ball Room)*

H. T. BARKER, Bird & Son Inc., E. Walpole, Mass., *presiding*

*Symposium on Fiber Shipping Case Performance*

1. "An Unscientific Way to Scientific Help in Packaging," by C. S. Macnair, Acme Steel Co. Discussion by W. J. Balster, Don L. Quinn Co.
2. "Some Aspects of Container Handling in Rail Transportation," by E. J. Dahill, Association of American Railroads.
3. "Quality Control of Corrugated Shipping Cases from a Buyers Viewpoint," by E. H. Balkema, Colgate-Palmolive-Peet Co.
4. "Controlling Container Quality," by George Farrah, Container Corp. of America.

## THURSDAY, FEB. 23, 1950

*9:15 a.m. Semichemical Pulping (West Ball Room)*

R. M. DRUMMOND, International Paper Co., Glens Falls, N. Y., *presiding*

1. "Penetration of Neutral Sulphite Cooking Liquors into yellow Birch Wood," by V. J. Wirpsa, St. Regis Paper Co. and C. E. Libby, N. Y. State College of Forestry.
2. "The Manufacture of Chemigroundwood Pulp from Hardwoods," by C. E. Libby and F. W. O'Neil, N. Y. State College of Forestry.
3. "Effect of the Presence of Bark in Semichemical Pulping," by E. L. Keller, Forest Products Laboratory.
4. "Effect of Mild Treatments with Sodium Hydroxide in Producing High Yield Pulps from Aspen," by K. J. Brown and J. N. McGovern, Forest Products Laboratory.

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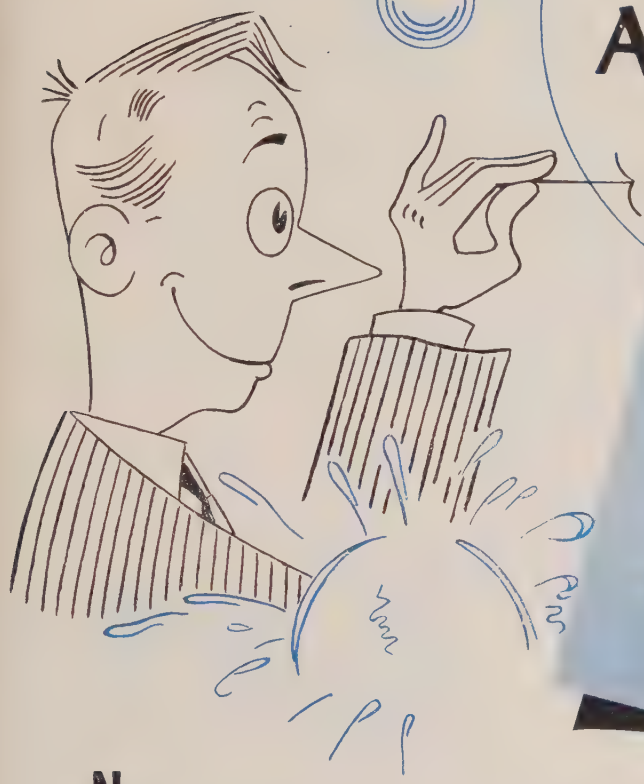

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5. "Groundwood Pulping of Mixtures of Spruce and Aspen," by Axel Hyttinen and E. R. Schafer, Forest Products Laboratory.
6. "Quality Improvement of Deinked Stock by Centrifugal Separators," by H. P. Bailey, J. J. Forsythe, and J. P. Tomaszewski, International Paper Co.

***9:15 a.m. Acid Pulping (Room B-C)***

G. H. McGregor, Minnesota and Ontario Paper Co., International Falls, Minn., *presiding*

1. "Relation Between Yield and Properties of Spruce and Aspen Sulphite Pulps," by J. N. McGovern, Forest Products Laboratory.
2. "Ammonia-Base Sulphite Pulping of Hardwoods," by E. F. Thode and Y. H. Lee, University of Maine.
3. "Proven Methods of Screening Maximum Quantities of Quality Pulp," by H. F. Schenk, Magnus Metals Corp.
4. "Circulation Systems and Sulphite Pulping," by J. M. Wilcox, Electric Steel Foundry.
5. "Use of Sulphite Waste Liquor in Cement," by E. W. Scripture, Master Builders Co.

***9:15 a.m. Chemical Products (Room A)***

Arthur Pollak, Consulting Engineer, New York, N. Y., *presiding*

Program on Tall Oil to be announced

***12:45 a.m. Annual Luncheon***

A. E. Bachmann, *President* of TAPPI, *presiding*

1. Presentation of the TAPPI Medal to George H. Mead, Mead Corp., by Reuben B. Robertson, Champion Paper and Fibre Co.
2. "The Marshall Plan and the Pulp and Paper Industry in Europe," by Joseph E. Atchison, Pulp and Paper Division, Economic Cooperation Administration, Washington, D. C.

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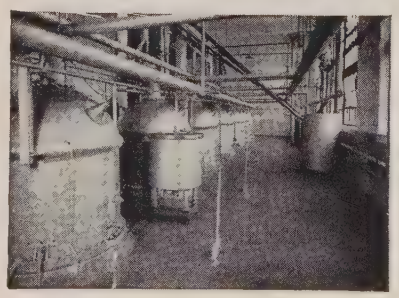

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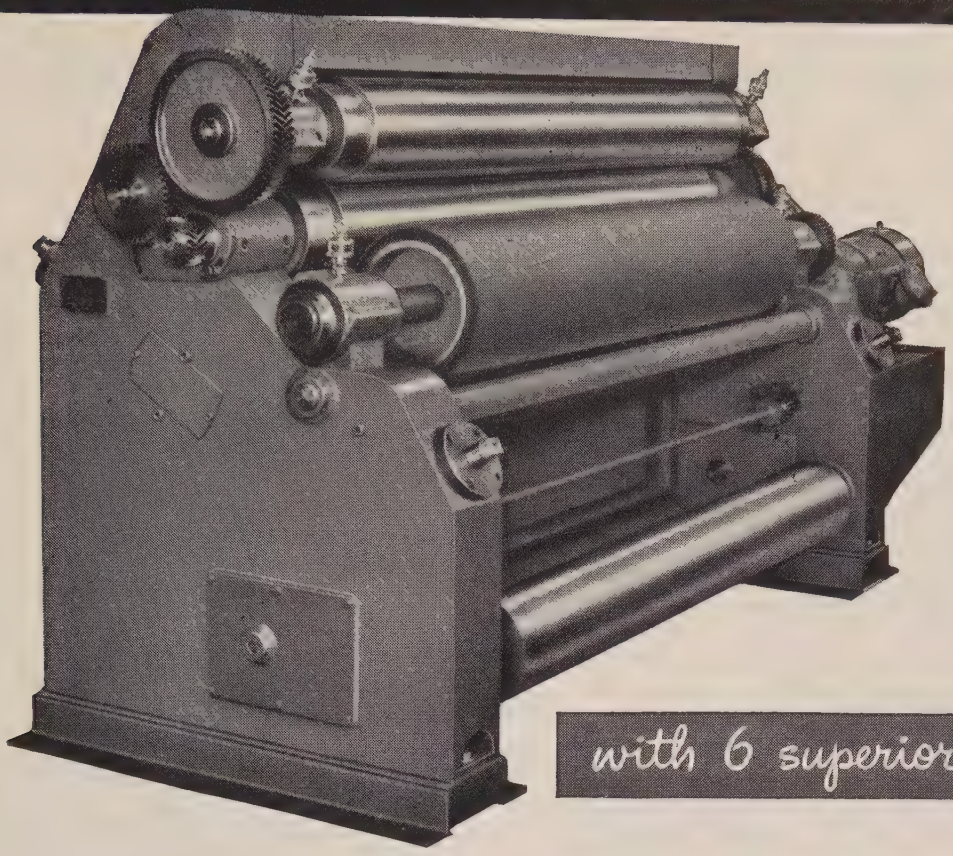

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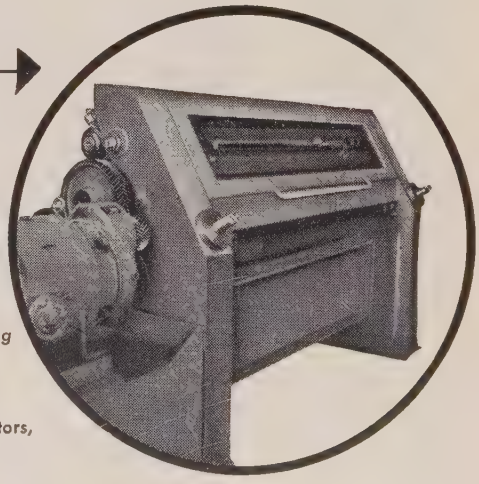

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# Present and Potential Utilization of Hardwoods in the Pulp and Paper Industry of Western Europe

JOSEPH E. ATCHISON

DURING THE period April 10, 1949 to Sept. 10, 1949, the writer made a survey of the present and potential use of hardwoods for the production of pulp and paper in seven Marshall Plan countries. Countries visited were: Italy, France, Western Germany, Austria, Belgium, Holland, and the United Kingdom. Information was obtained by discussions with Trade Association representatives, industry executives and technical men, Government representatives, ECA officials in the country missions, and by visiting numerous mills, pulp and paper schools, and development laboratories in the various countries. The following report summarizes the information obtained and the observations made on the use of hardwood.

In view of the limited amount of softwood pulpwood available in Western Europe, it is believed that extensive development of the use of available hardwoods as a source of fibrous raw material can become of considerable importance to the pulp and paper industry of several of these countries. In many countries where pulpwood supplies are limited, large scale cultivation of fast-growing trees such as poplar offers tremendous possibilities for the future.

## DEVELOPMENTS IN ITALY

The principal use of hardwoods in Italy is for production of mechanical pulp for use in newsprint and other printing papers. All but a very small percentage of the mechanical pulp manufactured in Italy is produced from poplar. In addition, a small amount of spruce from the Italian Alps is made into mechanical pulp.

For the past twenty years, poplar has been grown in Italy as a crop. The Experimental Institute for Poplar Cultivation, owned by Cartiere Burgo, has worked on development of improved strains of poplar trees which are resistant to decay and to all tree diseases. The institute grows great quantities of small trees which are distributed to the peasants for planting and cultivation on their own small plots of ground. This program has proved to be highly successful. The trees grow to pulpwood size in 10 to 12 years and have proved to be a nice source of income for the peasants.

Because of the great demand for wood for other purposes, the cost to the mills is extremely high, being about $36 to $40 per cord as compared to $16 to $20 in the U. S. and Canada.

Total production of poplar mechanical pulp is now approximately 500 metric tons per day or about 140,000 metric tons per year. Almost every mill in Italy, which produces newsprint or other printing papers, has its own grinding equipment for production of poplar mechanical pulp.

JOSEPH E. ATCHISON, Member TAPPI, Chief, Pulp and Paper Section Forest Products Branch, ECA, Washington, D. C.

The Burgo Co., in its development laboratories and pilot plant, has conducted extensive experimental work on a high-yield semichemical process for production of poplar pulp of greater strength than groundwood. They spray the chips with a low concentration of chemical, then defiber the chips in an attrition mill which they have designed and built themselves. By this process they have produced a poplar pulp in 90% yield which has many of the qualities of groundwood but which has far greater strength. They hope to be able to make newsprint with 100% poplar, thus eliminating the need for unbleached sulphite softwood pulp, by blending this semichemical pulp with their regular mechanical poplar pulp in about a 50–50 ratio. They feel certain that they can, at least, reduce the unbleached sulphite pulp content to a considerable extent.

The same company also uses a limited quantity of poplar for production of unbleached sulphite pulp which they blend with their softwood sulphite in the production of newsprint.

The only sizable use of hardwood for chemical pulp is by Snia Viscosa for the production of rayon pulp. This operation is described below.

A limited quantity of rough structural board is being produced from extracted chestnut chips, but these are not being used for production of any chemical pulp at the present time.

There are no mills in Italy now producing bleached semichemical pulp from hardwoods, although a limited amount of experimental work is now being carried out along this line. Many mills were extremely anxious to learn of the highly successful use of the neutral sulphite semichemical process in the United States for production of bleached pulp. All available information on this development was passed on to them, including cooking conditions, refining procedures, equipment used, and possible end uses of the product.

In summary the extent of the use of hardwoods in Italy at the present time is estimated as follows

| *Product* | *Production per day, metric tons* | *Production per year, metric tons* |
|---|---|---|
| Mechanical pulp from poplar | 500 | 140,000 |
| Unbleached sulphite pulp from poplar | 5 | 1,500 |
| Rayon pulp from beech | 85 | 25,000 |
| Structural board from extracted chestnut chips | 20 | 6,000 |
| Total | 615 | 172,500 |

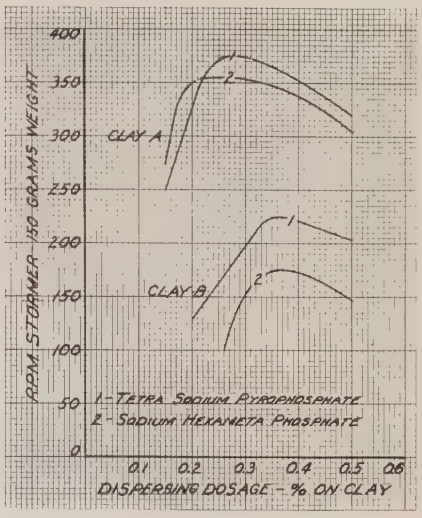
### *Production of Rayon Pulp from Beechwood*

Snia Viscosa, the largest artificial textile manufac turer in Italy and the only producer of rayon pulp, manufactures about 85 metric tons of rayon pulp per

# KAOLIN CLAY FACTS... *for Paper Makers*

NUMBER 1 OF A SERIES FROM THE J. M. HUBER CORPORATION LABORATORIES

**CHOICE OF DISPERSING AGENT IMPORTANT IN HIGH CONCENTRATION CLAY-WATER SYSTEMS.** In operations requiring fluidity in highly concentrated clay-water systems, the selection of the right dispersing agent in the proper quantity is extremely important. Dispersing agents in common use for such purposes are the complex poly-phosphates, such as sodium hexametaphosphate, sodium tetraphosphate, or tetrasodium pyrophosphate. In some cases, the addition of small quantities of strong alkalies, such as soda ash or caustic soda, along with the phosphates produces greater liquification than can be produced by the use of a single material.




Effect of concentration of dispersing agents on flow properties of a 70% clay-water system. (Graph shows two Huber Special Hydratex Clays.)

Determining clay brightness at the Huber Clay Laboratories.

**"AERFLOTED" FILLER CLAYS CUT FREIGHT COSTS 3-5%.** The cost of freighting filler grade paper clays has been cut up to 5 per cent in many paper plants by the use of Huber's "Aerfloted" clays. Secret is the lower moisture content of "Aerfloted" filler grade clays. They contain only 1 to 2 per cent moisture, while water washed fillers average 4 to 7 per cent. Use of water washed fillers, with their high moisture content, adds 3 to 5 per cent to freight cost.

| Per Cent Moisture in Filler Grade Paper Clays | |
|---|---|
| Huber "Aerfloted" Hi-White (Ga.) | 1-2 |
| Huber "Aerfloted" Paragon (So. Car.) | 1-2 |
| Huber Water Washed Filler (Ga.) | 3-5 |
| *Water Washed Filler "A" | 5 |
| *Water Washed Filler "B" | 4 |

(*Samples "A" and "B" were obtained from mills using these grades regularly, and are believed to be representative.)

**CLAY BRIGHTNESS UP AS MOISTURE DECREASES, STUDY SHOWS.** The final brightness value of paper grade clays increases as the moisture content decreases, recent experimental work at the Huber Clay Laboratories, Huber, Ga., reveals. With moisture content of 8.0 per cent, brightness of 82.1 per cent was recorded. With moisture reduced to 1.5 per cent, brightness jumped to 84.1 per cent. The work is reported in "Brightness Determination of Paper Clays," by N. Millman, Technical Director of the Huber Clay Laboratories. Reprints may be obtained by writing the J. M. Huber Corporation on your company letterhead.

"Aerfloted" Clay is passed through dryer at Huber plant to reduce moisture content.

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day from beech pulpwood. This wood is all imported from Yugoslavia. The pulp mill is extremely modern and a very high-quality dissolving pulp is being produced. It was laid out and built after the Snia Viscosa engineers had made a thorough study of United States and Swedish pulp production methods.

The beechwood is barked by hand because none of the barkers thus far tried have proved successful in getting the wood sufficiently clean. The mill manager had been investigating the possibilities of using a hydraulic barker, such as the Allis-Chalmers stream barker, for this purpose. Beech is extremely difficult to bark properly because of the close adherence of the bark to the wood, and the presence of an underlayer which is almost impossible to remove by ordinary barking methods. It was noted that the wood was cleaned completely in the wood room and every dark spot was removed. In contrast, mills using beechwood in some of the other countries did not do a complete job of cleaning the wood and as a result the dark spots carried through to the finished pulp.

The clean beechwood is then chipped in the conventional manner and cooked by the calcium bisulphite process in large stationery digesters, from which the pulp is blown into blowpits. This unbleached pulp is then run over knotter screens, washers, rifflers, centrifugal screens, flat screens, and thickeners, during which process it is cleaned rather thoroughly. It is then bleached in a multistage bleaching and refining operation. It is first chlorinated, then washed and run through a 10% caustic extraction stage, washed again and given a hypochlorite bleach. After a final washing, it goes to stock chests from which it is drawn and run onto a fourdrinier-type pulp forming machine and made into sheets about 80% moisture-free for use in the rayon mills.

The yield is about 38 to 40%, based on the moisture-free weight of the pulpwood.

***Need for Steam Economy***

Because of the high cost of steam and power, the engineering staff has been studying the possibility of concentrating and burning their waste sulphite liquor merely for its fuel value. They have determined that this would be an economical operation even if heat alone were recovered. At the writer's suggestion, they plan to investigate the new Weyerheuser sulphite pulping operation using magnesia base liquor in which both heat and chemicals are recovered.

This company has also conducted extensive experiments on the use of eucalyptus for production of rayon pulp. In runs of 1,000 tons, they have found that this wood gives a superior product for high-quality rayon. It is possible to grow the eucalyptus trees in Italy to pulpwood size in 8 years. An additional advantage is the fact that with one planting as many as five crops can be obtained from a single tree, thus replanting is necessary only after 40 years. The company has concluded that raising eucalyptus as a crop for production of rayon pulp will be economical and they have proposed a project for large scale cultivation of this tree in Sicily. This project would eventually involve construction of a dissolving pulp mill in Sicily and could prove to be of great advantage to that island in creating an entirely new industry there.

## DEVELOPMENTS IN WESTERN GERMANY

The use of hardwoods by the pulp and paper industry in Western Germany is confined almost entirely to the production of dissolving pulp, principally for the rayon industry. Almost all of the dissolving pulp mills in the Western Zones use beech pulpwood from the large beech forests in these zones. Both the sulphite and sulphate processes are used for production of dissolving pulp from beech although the sulphite process is the more widely used.

Previous to the war, these mills produced an excellent quality dissolving pulp from beech but at the time of the writer's visit, the quality of the pulp being produced was far below prewar standards and far inferior to the dissolving pulp being produced in Scandinavia and North America. This poor quality is not, however, the fault of the wood but is due to poor production methods in the mills themselves.

It is believed that there is an adequate quantity of beech pulpwood in Germany to satisfy the total requirements of the dissolving pulp mills. Some of the mills now claim that they can produce a higher quality rayon pulp from imported spruce wood but it is believed that this claim is merely because of the fact that imported wood is actually less expensive to the mills than domestic pulpwood. In view of the plentiful supply of beech available, and the high quality of rayon pulp which can be produced from it, there seems to be no need for importation of any pulpwood for this purpose.

The German mills have done extensive experimental work on the production of rayon pulp from beech by various processes including the sulphite and sulphate processes and by means of prehydrolysis followed by the sulphate process. Although the yield of pulp was found to be higher when using the sulphate process, the total yield from the wood, including byproducts, was higher when using the sulphite process. Although the yield of dissolving pulp from beech by this process is only about 40% the over-all yield from the wood, including the yeast produced from the sulphite waste liquors, is about 70 to 75%.

In observing the operations of the dissolving pulp mills, it appeared that they do not have adequate cleaning facilities for either the wood or the pulp. This is, no doubt, one important factor responsible for the inferior quality of their pulp. The wood is not cleaned completely before chipping and as a result dark specks remain in the chips. Then after the chips are cooked by the sulphite process, the pulp is washed in pits, passed over rough screens, and on to the bleach plant. These screens are entirely inadequate to remove all of the dirt from the pulp, thus it goes to the bleach plant in a rather dirty condition.

In the bleach plant the unbleached pulp is run through an acid chlorination stage in which 30 to 40% of the chlorine demand is added. It is then washed, and run through a calcium hypochlorite stage in which an additional 30 to 40% of the chlorine demand is added. It is washed again and run through a second hypochlorite stage in which the remaining 30% of the chlorine demand is added along with about 1% caustic soda based on the dry weight of the pulp. The bleached pulp is washed again and given a stabilizing treatment with sulphur dioxide gas. Total chlorine demand amounts

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to about 3.5% based on the oven-dry weight of the pulp.

The bleached pulp is run over one set of flat screens and on to the stock chest from which it is drawn to the pulp forming machine. It was noted that the pulp was still dirty when it was run over the forming machine and admittedly was of poor quality. The reasons for this dirty pulp were undoubtedly the inadequate screening capacity, the absence of rifflers in the pulp system, and inadequate wood-cleaning equipment.

In contrast to this operation, the Italian dissolving pulp mill, using the same type wood, produces an extremely clean pulp of superior quality. In order to meet this quality, it appeared that the German mills would have to install completely new and adequate cleaning systems.

No important use is being made of hardwoods for paper pulp at the present time. The pulp and paper school at Darmstadt has done experimental work on the use of hardwood for paper pulp. However, since beech is the only hardwood available in Germany in large quantities, and pulp produced from it by the conventional processes has poor quality as a paper pulp, the commercial use of hardwoods for paper has not been given much consideration.

It seems very possible that the neutral sulphite semichemical process might be applied to beech with much better results than has been obtained by the conventional pulping processes. Many hardwoods are being used successfully for production of paper pulp by the semichemical process and by modifications of the conventional processes. It would certainly be worth while to investigate these possibilities in Germany.

One development which may become of tremendous importance to Western Germany in the future is the cultivation of poplar for use as pulpwood. Dr. Jayme at Darmstadt has been conducting extensive studies in this field. He has several small poplar plantations on which he is growing many strains of poplar to determine the most suitable type for cultivation in Germany. Thorough studies are made on each type as to rate of growth, cellulose content during various stages of growth, resistance of plant diseases and decay, soil conditions most suitable for growth, etc. In addition to these plantations, some poplar has been planted in the forests on an experimental basis.

Thus far, the strains of poplar studied have been found to require soil with rather high moisture content. Thus originally, it will be planted along streams and rivers or in small areas of the forests where the moisture content is high. However, they hope to be able to develop strains of poplar which will grow in drier areas so that more extensive cultivation of this tree will be possible.

Because of the extremely rapid growth rate of poplar and the variety of its uses for pulp and paper, this development may prove to be extremely important to the German pulp and paper industry. The industry has realized the importance of the possibilities, and as a result no less than five associations are now in existence in Germany to promote the growing of poplar.

Poplar trees grow to good pulpwood size in 10 to 12 years as contrasted to 50 to 60 years for spruce and 80 to 100 years for beech, the principal pulpwood species now being grown in Germany. Poplar pulpwood could be used for all of the pulp now being produced from beech and for a large quantity of the pulp now being produced from spruce. Thus if large scale cultivation of poplar is successful, the growing cycle may be reduced drastically from the present 80-year cycle.

Dr. Jayme has found that the cellulose content of poplar reaches a maximum at an age of 10 to 12 years at which time it is of excellent size for pulpwood. The cellulose content goes down at an age of about 20 years because of the higher proportion of lignin present at that time.

Dr. Jayme has proposed a plan to plant poplar extremely thick, then after 10 to 12 years take out about 85% of the trees for pulpwood, leaving 15% to mature for use in producing matches, crates, and other wood products. The German Forestry Department seems to be opposed to his plan because they are not interested in growing trees specifically for pulpwood. This results from the fact that the top price for pulpwood is about 50 marks per raummeter as compared to about 200 marks for saw timber. They are against cutting the young trees for pulpwood but always want to leave all of them to mature to saw timber size. Dr. Jayme argues logically that by growing poplar in this manner, it will be possible to save large amounts of spruce and beech now being used for pulpwood and, in addition, imports of highpriced pulpwood can be decreased. This plan would certainly be of great benefit to the pulp and paper industry of Germany and should be given every possible consideration.

While investigating possible fast-growing woods for cultivation in Germany, Jayme studied eucalyptus in Australia. It was found that this tree does not grow well in Germany. In addition, the fiber does not have as much strength as poplar. The Australians have actually been replacing their eucalyptus with fast-growing pine trees with great success in recent years. They have obtained yields of 250 to 500 cubic feet per acre per year by using the same planting procedure as advocated by Jayme for poplar in Germany. Trees are planted very thick, then at about 15 years of age the small trees—about 6 inches in diameter—are cut for pulpwood. Then the remaining trees are left until about 40 years of age to be cut for saw timber. From the young pine trees they are able to produce a beautiful white groundwood pulp as well as chemical pulp for use in newsprint. Australia now has about 200,000 acres planted in pine and many farmers have found that growing it as a crop has proved to be very profitable.

Growing of poplar as a crop has been very successful in Italy and it is certainly probable that it could be equally as successful in Germany.

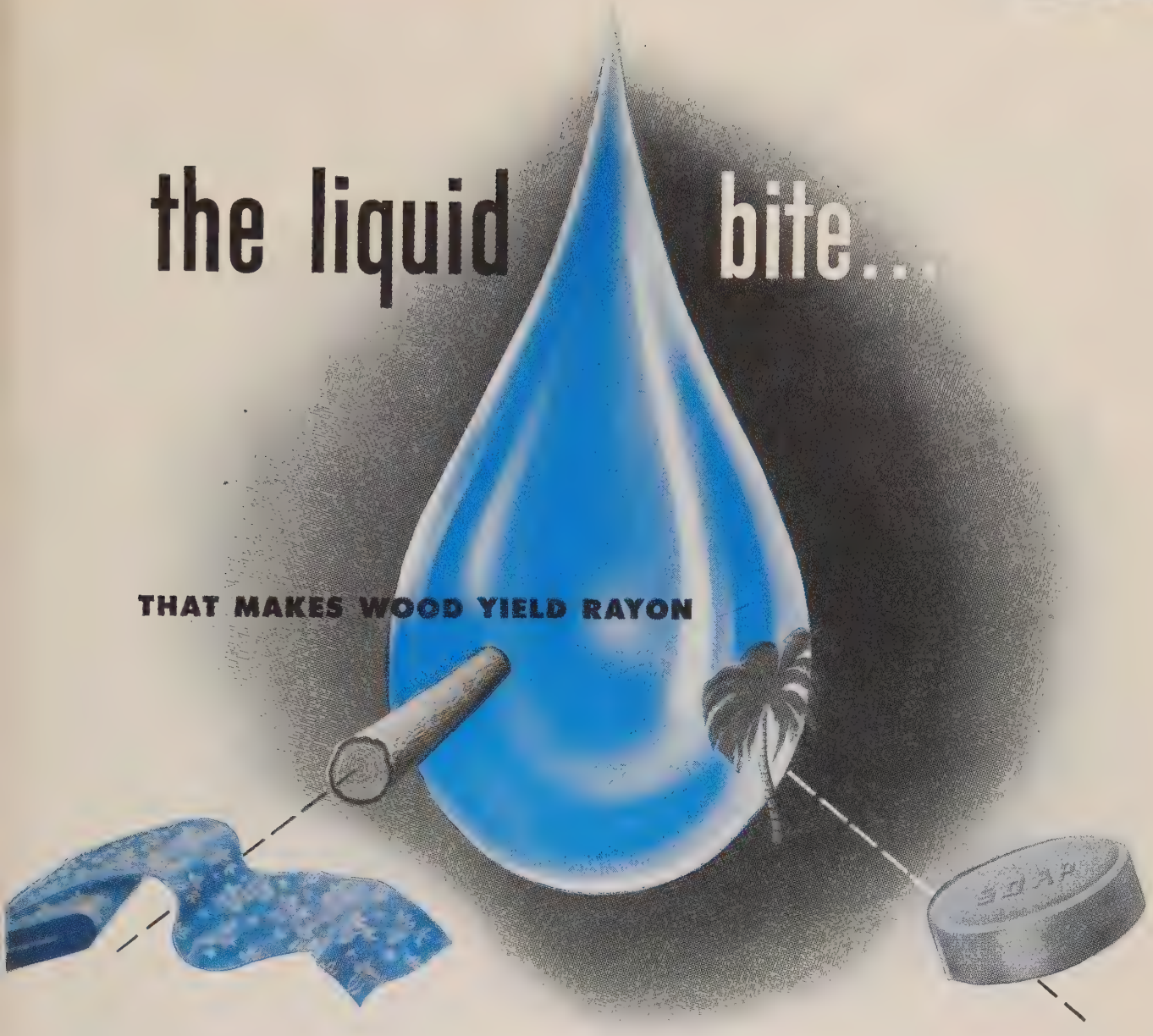
### DEVELOPMENTS IN BELGIUM AND THE NETHERLANDS

Since there is practically no pulpwood of any kind available in the Netherlands, there has been no important development in the use of hardwoods for pulp and paper.

The use of hardwoods in the Belgium pulp and paper industry is confined to rough grades of products such as wallboard and roofing felt.

There is one wallboard mill in Belgium which uses waste from match factories supplemented with low-grade poplar and birch as their fibrous raw material.

# the liquid bite...

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They use the Asplund defibrator process for production of about 25,000 tons of wallboard per year from these materials.

Papeteries General also uses the Asplund process for preparation of about 4000 metric tons of pulp per year for use in their roofing felt which is used as the base stock for floor covering. They also use lowgrade poplar and birch pulpwood as their fibrous raw material.

At the time of the writer's visit, no use was being made of hardwoods for the production of high-grade pulp or paper. Since pulpwood supplies in Belgium are extremely limited, it is doubtful that there will be any appreciable development in this field in the future.

## DEVELOPMENTS IN FRANCE

At the present time, hardwoods are used on a very limited scale for the production of pulp and paper in France. However, a number of mills are conducting experimental work on the use of the semichemical process for the production of increased amounts of pulp from hardwood. Expansion of production facilities for producing semichemical pulp are being held up by several mills at the present time because of the waste liquor problem. No satisfactory system has been worked out for recovery of the waste liquors and most mills are located where they would not be allowed to run this liquor into the streams. Thus, the difficulty of handling the waste liquor seems to be the major obstacle in the way of the expansion of the use of hardwoods and high yield pulping processes for softwoods. Some work is being done at the school in Grenoble and at a few of the mills on this problem although no intensive program seems to be in progress.

Most of the mills which are now using hardwoods were visited, and discussions were held with mill executives, technical directors, and production men in these mills. Summary reports were left with each of them regarding the progress which has been made in the United States in the use of hardwoods and straw. In addition, contents of these reports were discussed and many questions were asked by these men regarding the processes used, the best type of equipment for these processes, yield to be expected, quality of pulp obtained, costs of production, prospective uses of the pulp, etc. During this time, the plans which these and other companies have for future improvements and increased production capacity were revealed, thus giving some rather good background information on developments to be expected in the French pulp and paper industry.

Total production of pulp from hardwoods at the present time in France is no more than 100 metric tons per day or 30,000 metric tons per year. Of this amount, about 20,000 metric tons consists of bleached soda pulp made from extracted chestnut chips. Three different companies, who are primarily engaged in the chemical industry, operate these soda pulp mills as a secondary operation. Each produces about 6500 metric tons of bleached pulp per year. One of these mills converts its entire production into dissolving pulp of high alpha-cellulose content. This is the only dissolving pulp being made in France at the present time. Another of these mills operates a book paper machine and uses the bleached chestnut pulp in the book paper along with bleached softwood sulphite in a 80–20 ratio. Although this company makes various grades of book and printing paper, they always use 80% bleached chestnut pulp in the furnish.

There is a small amount of hardwood being ground into mechanical pulp for use in printing papers and in boards. One company is producing a rather strong groundwood pulp from birch by steaming the logs before grinding them. Total production of groundwood from hardwood is approximately 20 tons per day or 6000 tons per year.

Only one company thus far has completed facilities for large scale production of semichemical pulp from hardwood. This company, La Rochette, has installed equipment for production of about 60 metric tons per day but because of lack of power, their present production is limited to about 10 to 12 tons of semichemical pulp per day. All of this pulp is used in paperboard for boxes. In addition to using hardwoods for high yield pulp, they also use a semichemical process for obtaining high yield pulp from softwood. This pulp is also used in their paperboard. Several other mills are also conducting experimental work on the use of semichemical hardwood pulp for paperboard.

There was no indication of any work being done on the use of bleached hardwood semichemical pulp for high-grade papers. There has been a rapid development in this direction in the United States in recent years and it appears to offer an important field for development in France. Bleached hardwood semichemical pulp can be used in small amounts in almost all grades of papers and in large amounts in some grades which are now being produced entirely from softwood pulps.

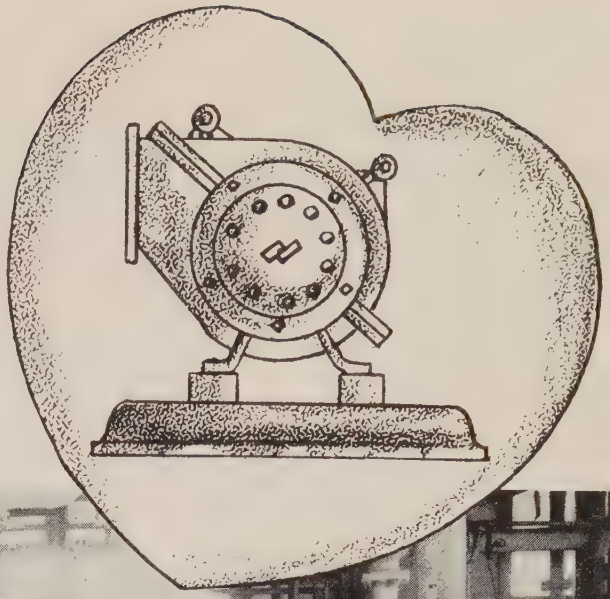
Summary of estimated production of pulp from hardwoods in France during 1948 is as follows:

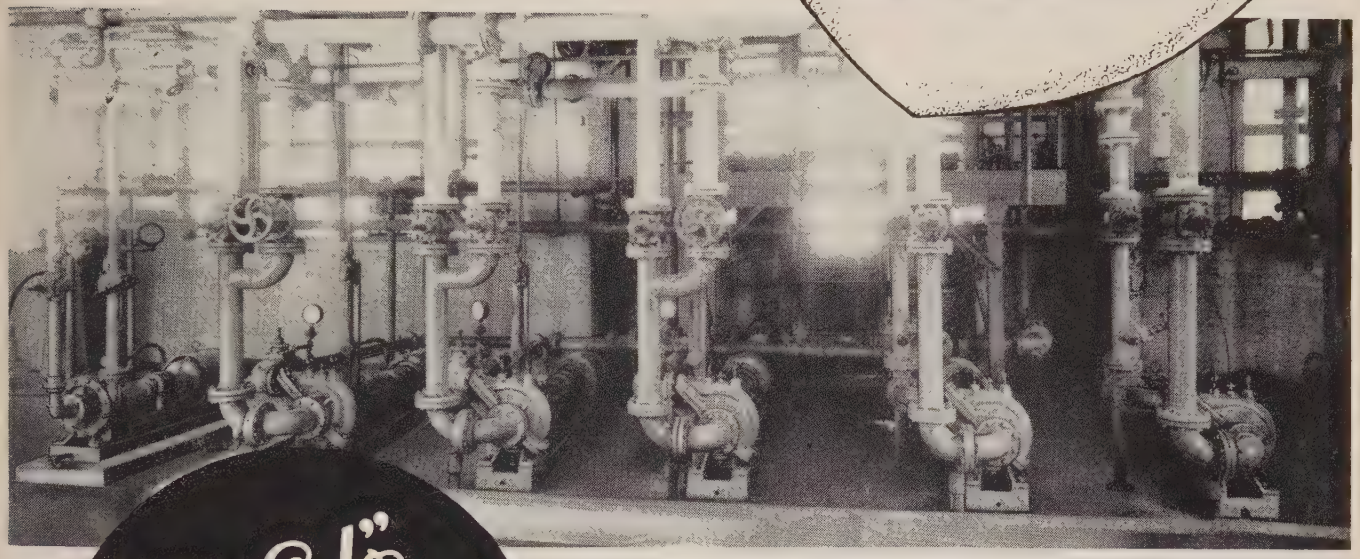
| *Grade of product* | *Amount, metric tons per year* |
|---|---|
| Bleached soda pulp from extracted chestnut chips (of which 5000 tons were for dissolving pulp) | 20,000 |
| Mechanical pulp from poplar and birch | 6,000 |
| Semichemical pulp for paperboard | 4,000 |
| Total | 30,000 |

In view of the extreme shortage of softwood pulpwood in France and its dependence on imports to supply existing pulp mills, it is believed that there will be extensive developments in the use of hardwoods by the pulp and paper industry in France.

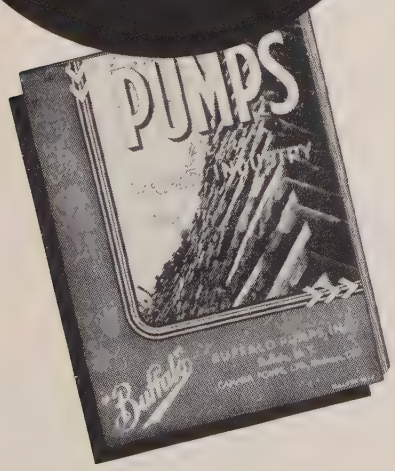
It is possible that large scale cultivation of fast growing trees such as poplar for use as pulpwood might be equally as successful in France as in Italy. Poplar can be grown to pulpwood size in 10 to 12 years as contrasted to 50 to 60 years for spruce and pine, the principal species now being used. It could be used extensively for the production of pulp and paper, and the forest area necessary for sustained yields of poplar pulpwood would be far less than is necessary for these softwoods. As mentioned previously in this report, Italy has been very successful in this field and, at the present time almost 100% of the mechanical pulp used for newsprint in Italy is produced from poplar.

# When the Pump is the "HEART" of the Process-





"Buffalo" PAPER STOCK PUMPS

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### *Production of Bleached Soda Pulp from Extracted Chestnut Chips*

There are three chemical companies in France which have gone into the manufacture of pulp merely to make use of a byproduct, the chestnut chips remaining after extraction of the tannin from them.

These mills use practically the entire chestnut tree and break this wood into very small chips for extraction. For this purpose they use a rather unusual chipper which has knives on the periphery of a disk.

The chips are conveyed to extractors in which the tannin is extracted with low-pressure steam. At the completion of this process, chips are dumped out of the extractors at about 65% moisture content. This high moisture content of the chips has presented something of a problem when they later cook them for pulp because it prevents proper impregnation of the liquor. This difficulty has been especially troublesome when they have used the Asplund process for cooking the chips with a low concentration of chemical. Experimental work was in progress with various kinds of presses and dewatering equipment in an attempt to reduce this water content to about 45% of the weight of the chips.

The extracted chips are screened in a rotary screen to remove fines which are burned, and oversized chips which can be further broken up. These chips are then fed to stationery vertical digesters in which they are cooked at about 120 pounds pressure by the soda process, using 17% caustic soda based on the dry weight of the chips. After cooking, chips are blown to diffusers and the pulp is washed. It is then run through a two-stage continuous bleaching process using chlorine and hypochlorite with no washing between stages. At completion of the chlorine stage, alkali is added for neutralization, then the pulp continues on to the hypochlorite stage. However, all of these mills plan to install washers between bleaching stages in the future.

Each of the three mills produces about 25 metric tons of bleached soda pulp per day by this process, or a total for all mills of 20,000 tons per year.

Part of this pulp is refined, in a variation of the bleaching process, for use as dissolving pulp. An excellent quality dissolving pulp of as high as 95% alpha-cellulose content has been obtained by this method.

### *Production of Semichemical Pulp by Use of Asplund Defibrator*

One of the mills mentioned in the section above has installed a complete Asplund defibrator unit for experimental production of about 10 to 12 tons of semichemical pulp per day using extracted chestnut chips as the raw material. This project was set up for the benefit of some of the company's smaller chestnut extraction plants in which the extracted chips have been burned previously. They are now in the process of determining the best cooking conditions, the best refining conditions, the best uses for the pulp so prepared, etc. The plan is to install defibrator systems in each of their two small extraction plants, to make use of the extracted chips in these plants. They have not decided as yet whether they will make wet lap pulp for sale or whether they will make board with the pulp themselves. However, from the results obtained thus far, the project seems certain of success and the pulp so prepared will doubtless have many uses.

At the time of the visit, they were adding about 5% caustic soda (based on the dry weight of the chips) to the chips in the chip bin. These were allowed to stand for some time until the cooking liquor penetrated, then the chips were run continuously into the Asplund defibrator cooking chamber where they were cooked at about 130 pounds pressure. After passing on through the defibrator disks, the pulp produced was of rather good quality. However, they plan to try many other cooking conditions and methods before going into commercial production.

### *Semichemical Pulp Mill at La Rochette*

The only mill which has completed facilities for production of semichemical pulp from hardwood is the La Rochette mill of Cartonnerie La Rochette. This company has a complete semichemical plant of modern design with a production capacity of 60 metric tons per day. However, because of lack of sufficient power, production was limited to 10 to 15 tons per day at the time of the visit.

The only part of the operation which appeared contrary to good practice was their failure to bark the wood before chipping it. This results in excessive use of chemicals, discoloration of the pulp and lower pulp strength. The unbarked wood is chipped in the usual manner and the chips are stored in chip bins above four large rotary globe digesters. These digesters, made in France, each hold about 7 to 8 tons of chips.

At the present time pine chips are being cooked by a modified soda semichemical process. They use 5% sodium hydroxide and 1% sulphur based on the dry weight of the chips, and cook at 80 to 100 pounds pressure for about six hours. After completion of the cook, the chips are dumped into a v-shaped bin and conveyed to Bauer refiners. The yield is about 70% of a brown-colored pulp which is used as bottom liner for their test liner board.

The pulp goes from the Bauer refiner to a chest from which it is drawn to a Swedish wet lap machine and made into wet laps for storage at about 50% moisture content. The only washing the pulp receives is on the vacuum thickener of the wet machine.

In addition to cooking pine, they also cook some poplar wood by the neutral sulphite semichemical process, using about 10% sodium sulphite and 4% soda ash based on the dry weight of the wood, cooking for 4 hours at 80 to 100 pounds pressure. This gives them a yield of only 63% of a light greyish-colored pulp. Normally, in the United States, yields of 75% are obtained by this process. The difference is, no doubt, due to the fact that the yield here is based on the weight of the unbarked wood and in the United States the yield is based on the weight of the bark-free wood. The color could also be improved greatly if the wood were barked before cooking.

This poplar semichemical pulp is used in place of old newspapers as filler furnish for their board or in the bottom liner furnish. It gives greater strength to the board than does waste news.

As soon as power is available this company plans to increase the semichemical production to 50 or 60 metric tons per day.

*Ready reference chart...*

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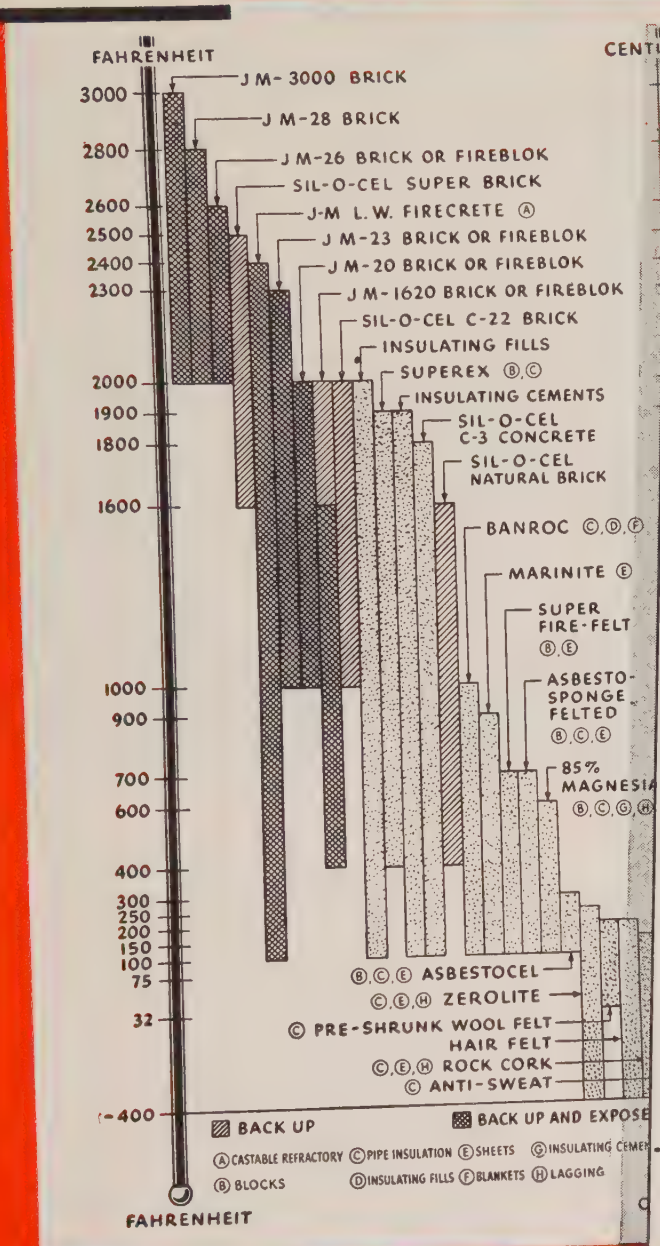


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## DEVELOPMENTS IN AUSTRIA

The use of hardwoods is limited to the use of beech pulpwood for production of dissolving pulp. There is only one mill using beech for this purpose and it is producing about 1500 metric tons of dissolving pulp per month. There are adequate quantities of beech pulpwood available in Austria for this purpose. It is believed that the use of beech for this purpose can actually be increased in view of the plentiful supplies of this wood which are available. The quality of the dissolving pulp being produced is far inferior to that being produced in Scandinavia and North America. However, this poor quality is not the fault of the wood but is because of poor production methods in the mills themselves.

No use is being made of hardwood for paper at the present time. Since beech is the only hardwood available in Austria in large quantities, and pulp produced from it has poor quality as a paper pulp, the commercial use of hardwoods for paper has not been given much consideration.

In view of the tremendous amount of rainfall in Austria, it seems probable that the cultivation of poplar for use as pulpwood could be highly successful. Poplar trees grow to pulpwood size in 8 to 10 years as contrasted to 50 to 60 years for spruce and 80 years for beech, the principle pulpwood species now being used in Austria. Poplar pulpwood could be used for all of the dissolving pulp now being produced from beech and for a considerable portion of the paper pulp now being produced from spruce, including mechanical pulp for newsprint. Thus, if large scale cultivation of poplar could be promoted, the growing cycle might be reduced drastically and the yearly yield of pulpwood could be increased greatly.

Because of the rapid growth rate of poplar, and the variety of its uses for pulp and paper, it is believed that the possibilities of cultivating it in Austria should be investigated thoroughly. Growing poplar as a crop in Italy has been very successful, and at the present time almost all of the mechanical pulp produced in Italy is made from poplar.

### *Production of Dissolving Pulp from Beech*

One mill in Austria produces about 1500 metric tons of dissolving pulp per month using beech pulpwood as a raw material. This mill is located beside a rayon and staple fiber plant and all of the dissolving pulp is sold to this plant. Both plants were visited by the writer and discussions were held regarding their mutual problems.

It was brought out in the discussions that the quality of the dissolving pulp produced in Austria has been poor and nonuniform. This has caused difficulties in operation in the staple fiber mills. Some of the reasons for this low quality of the dissolving pulp were observed when the pulp mill was visited. These observations were discussed with the mill staff and changes were pointed out in methods of operation and in additional equipment which would result in a considerable amount of improvement in the quality of the product.

One of the most obvious reasons for the low quality, just as in the German mills, appeared to be inadequate cleaning of both wood and finished pulp. The beechwood was not being cleaned sufficiently to remove all of the inner bark and dark spots before the wood went to the chippers. Then the pulp was not being cleaned properly either before or after the bleaching operation. They used no low consistency riffling operations to remove the heavy dirt and knots and the screening capacity was entirely inadequate to remove the lightweight dirt. Thus the bleached pulp came off the pulpforming machine containing large amounts of dirt specks and was far inferior in quality to the dissolving pulp produced in Scandinavia and North America.

It has been proved that excellent quality dissolving pulp can be produced from beech if proper methods and equipment are used. It is believed that the dissolving pulp mills in Austria will have to take steps to improve the quality of their pulp both for home consumption and in order to compete on the world markets. In addition to installation of equipment for proper cleaning, it is believed that improvement could be made in the bleaching and refining operation, especially the caustic extraction process, which would result in much higher quality pulp.

## DEVELOPMENTS IN THE UNITED KINGDOM

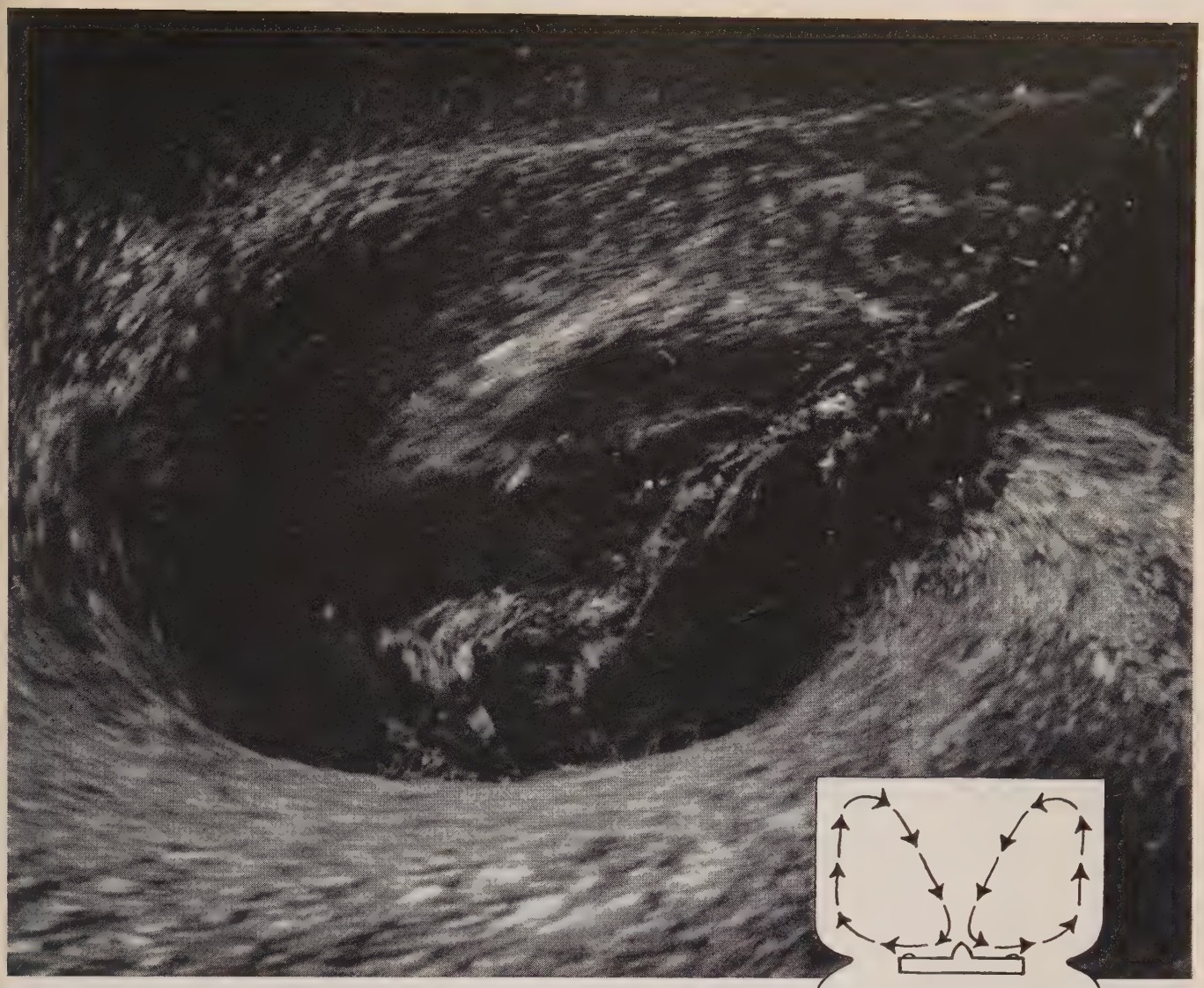
Although there is no domestic pulpwood available in the United Kingdom for production of high-grade pulp, there is some waste hardwood being used for production of wallboard. This consists of waste willow from diseased trees, and tree trimmings from the Forest Service lands. It is made into both insulating board and hard board.

The wallboard mill uses a furnish consisting of 50% Asplund fiber and 50% groundwood, both of which are prepared from the domestic waste wood mentioned above. The same furnish is used for both hard board and insulating board. Total production amounts to about 28 metric tons per day.

## DEVELOPMENTS IN THE SCANDINAVIAN COUNTRIES

Although the writer did not visit the Scandinavian countries on this trip, it has been reported by representatives of the Scandinavian pulp and paper industry that the use of hardwoods has been increasing greatly during the past few years.

Birch is now being used extensively by some mills in the manufacture of sulphate-type pulp. While bursting strength, tear, and other strength properties of finished paper manufactured from this pulp are not quite as high as those resulting from use of coniferous sulphate pulp, the birch pulp seems to be in considerable demand. It is currently being sold at $10 per ton below coniferous wood pulp which no doubt plays an important part in encouraging its use in as many papers as possible.



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# The Position of Cellulose as a Chemical Raw Material

E. A. TIPPETTS

THE SELECTION of the subject "The Position of Cellulose as a Chemical Raw Material" for discussion is, I believe, a very timely choice. Timely because the attack by synthetic polymers upon the position of cellulose in the plastic, fiber, and film fields has become greatly accelerated in the years since the last war. It is the purpose of this paper to review the effects of this attack, and if possible, predict some of its future effects.

Cellulose was the first fairly pure polymeric raw material available to the chemist in large quantity, and indeed, a very thriving chemical industry based on cellulose had emerged even before the chemical structure of cellulose was established and before the word "polymer" had much meaning to the chemist other than a descriptive term to describe reaction residues to be discarded in the waste jar. Cellulose has proved to be a surprisingly versatile polymer. Its versatility is aptly illustrated by the following paragraph from an anonymous writer in a recent trade publication of the International Nickel Company.

" . . . . a man can rise at dawn and apply brushes with nitrocellulose handles to his teeth and hair, eat a sausage breakfast complete with viscose casings, and ride forth into the country in a car finished with cellulose lacquers and provided with artificial leather seat from the same source. If he is impressed by the beautiful countryside as he views it through clear safety glass made with cellulose derivatives, he may step into the woods to take a few snapshots on nitrocellulose film. Suppose tragedy now intervenes and a hunter mistakes him for a browsing moose and shoots him with a bullet propelled by smokeless nitrocellulose powder! The victim can inform his wife that she is a widow by writing a note on linen paper that is nearly pure cellulose, and she will mourn him in a new rayon dress of the regenerated molecule."

Last year the chemical industry consumed about 750,000 tons of chemical grade cellulose. This consumption is very roughly double that of ten years ago. Notwithstanding the almost endless variety of cellulosic products turned out by the chemical industry, the major factor responsible for the growth of the dissolving pulp segment of the pulp field has been the growth of viscose and acetate rayon and cellophane. At present, roughly 85% of the chemical grade cellulose consumed in the United States is used in the manufacture of these products, with the remainder being roughly split 50/50 between plastics and miscellaneous products comprising smokeless powder, lacquers, fabric coatings, films, etc. Furthermore, there are strong indications that the importance of cellulose will decrease in the plastic and miscellaneous fields in the years immediately ahead.

Plastics are a good example showing the impact of synthetic polymers upon the position of cellulose as a chemical raw material. In the last three years the consumption of cellulosic plastics in the United States dropped 38%. In this same period total plastic consumption increased 27% due primarily to the rapid growth of polystyrene and vinyl plastics. At present cellulosic plastics account for less than 10% of the total plastic consumption.

Likewise in the miscellaneous fields of lacquers, photographic film, fabric coatings, etc., there is little reason to believe there will be any major increase of the consumption of cellulose in these industries. Synthetic polymers will probably be used in increasing amounts in these fields once dominated by cellulose. In the one field of smokeless powder for which cellulose is peculiarly adapted, the industry has already passed through its period of major growth. Carboxymethyl cellulose, a new outlet for chemical cellulose, while expected to find increasing application in such uses as an aid to synthetic detergents and as a textile and paper size, a thickening agent for food stuffs, probably will remain a minor factor in the consumption of dissolving cellulose. Thus, rayon and cellophane remain as the critical fields, the growth of which will probably determine the future growth of cellulose as a chemical raw material.

In these fields, which as stated above, account for 85% of the consumption of chemical cellulose, the future picture is somewhat more optimistic. Both of these industries are expected to grow in the immediate years ahead. In 1947 these two industries consumed about 564,000 tons of cellulose, and it is a good guess that by 1951 they will require around 800,000 tons.

The processes used in the rayon and cellophane industries provide considerable latitude in manufacturing fibers and films having a wide range of properties. This fact has accounted for the rapid and sustained growth as illustrated by the recent development of rayon for automotive tires and the earlier development of moistureproof cellophane. Today 25% of the rayon produced in the United States is consumed by the tire industry. Ten years ago tire rayon amounted to less than 3% of rayon production. A similar story could be told illustrating the effect of the moistureproof cellophane on the cellophane industry. The momentum already gained by these two products, tire rayon and moistureproof cellophane, will account for the increased consumption of chemical cellulose in the next few years.

To date the pulp industry supplying cellulose for rayon and cellophane has concentrated primarily on improved pulp refining methods leading to higher alpha pulps of improved color at an acceptable level of reactivity and price. It is probably safe to say that this type of pulp research and development will not be sufficient to sustain the growth of cellulose as raw material for these industries past the immediate future. The pulp manufacturers must look upon themselves as polymer manufacturers in strong competition with a

E. A. TIPPETTS, Assistant Director, Viscose Rayon Research, E. I. du Pont De Nemours & Co., Richmond, Va.

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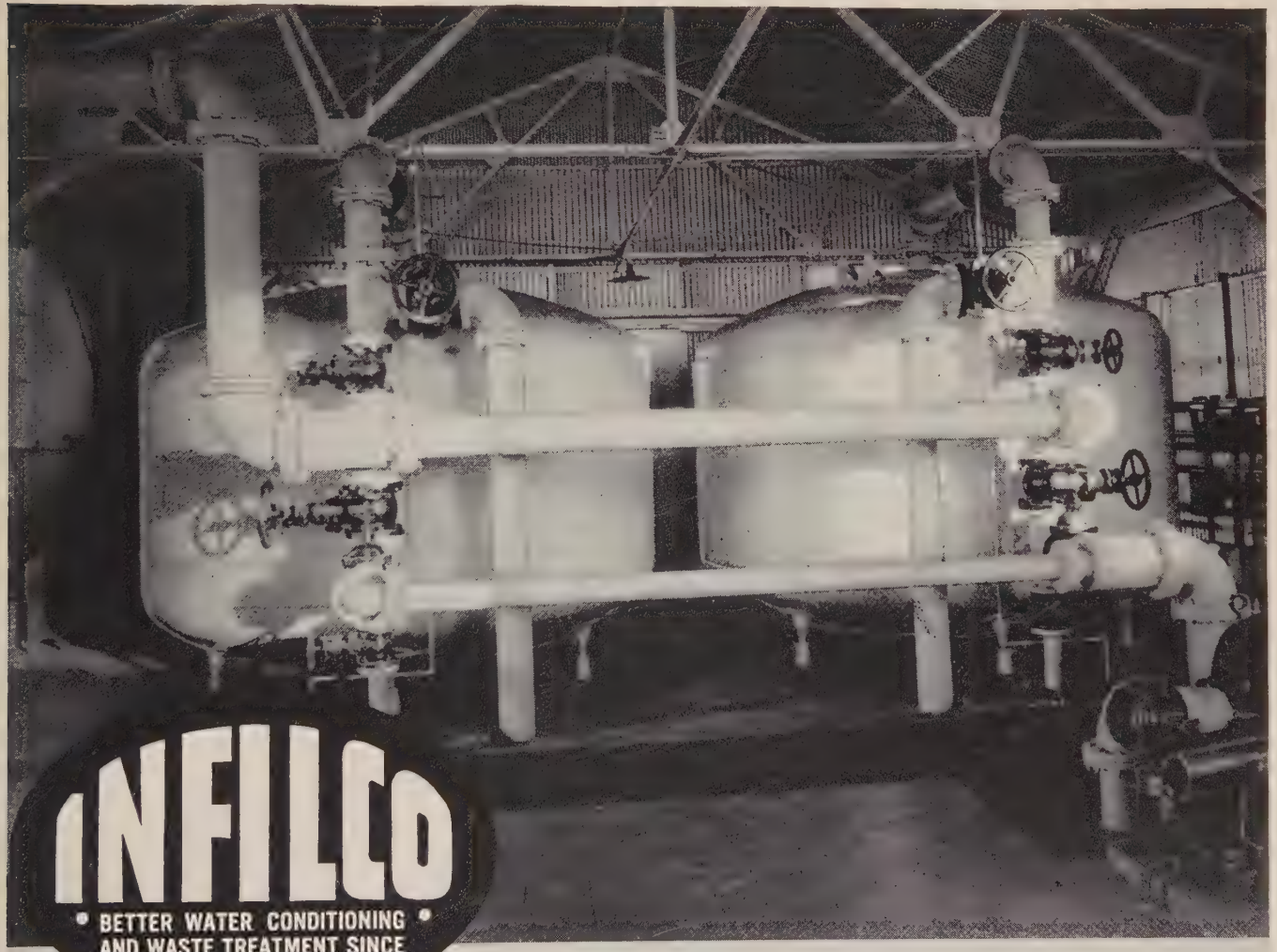

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dynamic synthetic polymer technology which is based on fundamental studies of the chemistry and physics of the processes and intimate knowledge of the effect of polymer structure on the final product. Nylon, "Orlon" acrylic polymer, and other man-made fiber and film polymers yet to come are and will be tailor-made with respect to purity, chain-length distribution, distribution of substituent groups along the chain and in other characteristics, to impart desired functional characteristics to the final product. Cellulose is still characterized in terms of strictly empirical tests. We do not know much about the effect of low levels of pentosan content on the functional properties of the resultant fibers or films nor do we know whether the traces of carboxy and aldehyde groups scattered along the polymer chain are deleterious or not. Neither is it known how a narrow chain-length distribution might affect the properties of fibers or films or the optimum physical state for the refined cellulose for maximum reactivity in the viscose or acetate processes. Nor do we know whether the cellulose chain is a polymer wholly composed of condensed beta glucose monomer units or whether somewhere in the chain there are not similar but significantly different carbohydrate groups. Answers to these and other equally important questions must be found and from such background should come the improved depolymerization and refining technology without which cellulose will find it increasingly difficult to compete with low cost synthetic polymers of the future. The answers to similar questions are known or are being rapidly obtained for synthetic polymers.

There are several reasons to believe that basic research programs answering these and other pertinent questions would yield profitable returns. It is known that some of the functional properties of rayon made from purified cotton linters are better than those made from wood pulp. This is particularly true for the tire cord rayon where tire cord fatigue-resistance obtained from yarns prepared from linters is significantly better than that obtained from the best commercially available sulphite pulps. In this instance, even though the chemical processes used by the rayon manufacturer are the same for both the linters and the wood pulp, some unknown intrinsic property of the cellulose derived from cotton linters carries through to the final product and significantly affects its functional behavior. This problem has received a great deal of attention in the past, but the answer is still obscure. I believe I am safe in saying that the difference is not wholly one of purity or of chain-length distribution. An answer to this question might well lead to modifications of the wood pulp refining process to result in a cellulose giving functional properties even superior to those now obtained from cotton linters cellulose.

Another indication that a basic study of cellulose refining might lead to improved celluloses, particularly for the rayon industry, is the fact that tire rayons made from "Cordicell,"* a wood pulp manufactured using kraft cooking following prehydrolysis, have properties better than obtainable with the best commercially available sulphite pulps and indeed approach closely the properties obtained using cotton linters. In this instance the standard cellulose characterization tests do not detect the reason why the prehydrolyzed kraft pulp should be superior to the sulphite pulp. Here again if the answer to this question was known, further improvements in pulp could probably be realized.

Thus far I have ignored economics. The chief virtue of cellulose as a polymer is its relatively low cost compared to other commercial polymers. This favorable position, too, must be maintained and if possible improved. As the synthetic polymer industry develops, this will be increasingly difficult. Already the competition of nylon with rayon is evident. During the recent textile market slump higher priced nylon textiles have sold while less expensive rayon goods have gone begging. While the reasons for this situation are extremely complex, certainly not the least important factor is the fact that on a functional basis nylon is beginning to successfully compete with rayon in value per dollar in some of the moderately large volume segments of the textile business.

The rayon manufacturers are keenly aware of this situation and are directing increasingly large proportions of their research effort to developing understanding of the fundamentals of their processes to aid in producing low cost cellulose yarns having improved over-all functional performance. In fact the major responsibility for keeping rayon competitive with the new synthetic fibers lies with the rayon manufacturers. I believe that the rayon industry will measure up to its responsibility and will make the best quality products possible from the raw material available for its use. However, the pulp manufacturer must recognize that an important part of the responsibility for improving the performance of rayon lies in his end of the business.

As regards cellophane, less is known of the importance of pulp characteristics on final product characteristics than is the case for rayon. Since many of the important functional properties of cellophane stem from the various coatings placed on the cellulose base film, there has been less need to investigate the importance of the characteristics of the base polymer on the functional properties of the final product. However, as new low cost polymers for films are developed, the problem of improving the cellulose base film of cellophane will become increasingly important. An improved cellulose polymer probably will also be required in this field.

In summary the position of cellulose as a raw material for the chemical industry simmers down to this: first, in all uses except rayon and cellophane there is little to indicate much continued growth. Second, in the fields of rayon and cellophane the use of cellulose will continue to grow for the next several years, largely capitalizing on technology already developed. Third, a broad fundamental study of pulping technology is urgently needed to hold and expand the volume of chemical cellulose against the low cost tailor-made polymers which will inevitably be made available to the fiber and film industries from the rapidly expanding field of basic research on synthetic polymers.

* A product of Uddeholm Actiebolag, Skoghall, Sweden.

# Mill Engineers' Selection of the Paper Machine Drive

ROLAND A. PACKARD

**A brief description is given of the paper machine of one hundred years ago. In those early days it was recognized that constant speed was of the greatest importance, but papermakers could not obtain it. Three so-called "rates of motion" were sufficient—high, medium, and low. In order to minimize the "great quantities of paper destroyed" due to uneven and changeable speeds required of the different sections of the machine, pieces of felt properly smeared with lagging wax were either applied or torn off. The Marshall train, introduced in 1890, made it possible to double machine speeds. When these speeds attained 600 f.p.m. the increased power demand roughened up the wooden teeth on bevel gears, which action developed vibration transmitted to horn couplings, thence to rolls and machine frames. At this time the V-belt made its appearance in power transmission so that short center application made it adaptable for drives. The hydraulic drive together with the hypoid and spherical bevel gears made their debut with the flat belt cone pulley combination. Description is given of each together with relative merits. Machine widths, speeds, and horsepowers, again began to tax the engineer's ingenuity, which brought out the individual motor on each section of the machine. This was a direct-current unit connected to a synchronous motor. All these motors were tied together electrically. Next came the synchronous regulators followed by amplified electronic systems of control. The development of yardsticks for the mill engineer is shown with a series of graphs relative to speed ranges for different groups of papers and a general selective guide for type of machine drive. The approximate cost per horsepower for different types of drive is given, together with the outstanding features of each type of drive. A number of illustrations complete the article.**

In the preparation of this paper the author has a primary responsibility to the paper-mill engineer and a secondary responsibility to the paper machine drive manufacturer. To help either one is to help the other.

The information to follow has come from personal experience as well as the results of research with other paper-mill engineers; also representatives of the manufacturers of the machine. It has produced some interesting and constructive conclusions. When the consensus of engineers shows certain trends it is only fair to manufacturers to try and point the way to an improved product toward the ultimate benefit of the financial balance sheets of all concerned.

## TYPES OF DRIVES

First, as a background to this treatise, the author would like to give a few excerpts from history, leading up to the time of his early experience in the operation of a paper machine. In 1866 this machine was described by A. Proteaux in his "Practical Guide for the Manufacture of Paper and Boards" as follows: "paper machines are classified according to the width of paper they are intended to make, the large size 75 to 104 inches; medium size 54 to 75 inches, and the small size 40 to 54 inches. The number of drier cylinders is variable; three is the minimum, five is the average, and some machines have as many as eight to ten driers. This last number seems to us exaggerated. Six appear to be the best number, of which four serve to dry the paper and two the felts.

"The paper machine, needing a perfectly regular motion, should have a motive power of its own, whether it be water wheel or steam engine. When the motive power to be disposed of is abundant, we advise the use of a small water turbine fed by a reservoir having its water always at the same level, and the reservoir may be placed at a considerable height, say in the upper story of the mill. Sometimes a small high-pressure engine is employed, and the steam, after being used as a motive power, serves for heating the drying cylinders.

"Each paper machine should be furnished with pinions admitting different velocities, or graduated pulleys, to allow the manufacture of thin or heavy papers.

"Three rates of motion are considered sufficient; the high, the medium, and the low. To facilitate the management of the machine it is well to have at each state of transmission, extensible pulleys, or those with variable diameters, as well as struts to prevent the belts from slipping."

Referring to drives and their troubles, the "Manufacture of Paper" written by Charles P. Dairs in 1886, states "great quantities of paper are destroyed by troubles with drives which are constantly occurring, often three or four times during a day's work, making it necessary that the relative speeds of the presses and drier cylinders should be frequently but slightly changed. The common way to accomplish this is by what is termed 'lagging.' A number of strips of canvas or felt or thick cloth about as wide as the face of the pulley, called 'lagging,' are kept on hand. When a change of speed is desired, one of these is smeared with lagging wax, so-called, and laid on the surface of the driving pulley to increase its diameter and quicken the speed of the pulley or gear at the other end. Through the number and length of the strips held on the pulley or belt by the wax, any slight change can be produced, and, although a very crude and clumsy expedient, this lagging has been in almost universal use up to the present time, as nothing practical, as yet, has been found to supersede it."

### *Bevel Gears*

The first real step in drive improvements came toward the end of the last century when the bevel gear idea was borrowed from the early water wheel

Roland A. Packard, Executive Vice.-Pres., Holyoke Machine Co., Holyoke, Mass.

corner drives. It was a simple addition to apply the cone pulley for each set of gears to handle the draw on each section of the paper machine.

***Marshall Train***

The cone pulleys were belted to a main shaft parallel and to the rear of the machine. This long shaft, in turn, had its speed varied by connection to two lengthy cone pulleys usually built with wooden faces to afford a differential spread of speeds for different weights and kinds of paper manufactured. A constant speed steam engine was the usual source of power. The mechanical power transmission, called the Marshall Train, born during the latter part of the last century, was such a boon to the papermaker that he felt little need for any change for a long time to come. Rope drives melded in with the Marshall drive and still may be found in use in some of the old mills. These bevel gear drives often were mixed with a maze of steam pipes, platforms, stairs, and belts. A typical view of this condition is shown in Fig. 1.

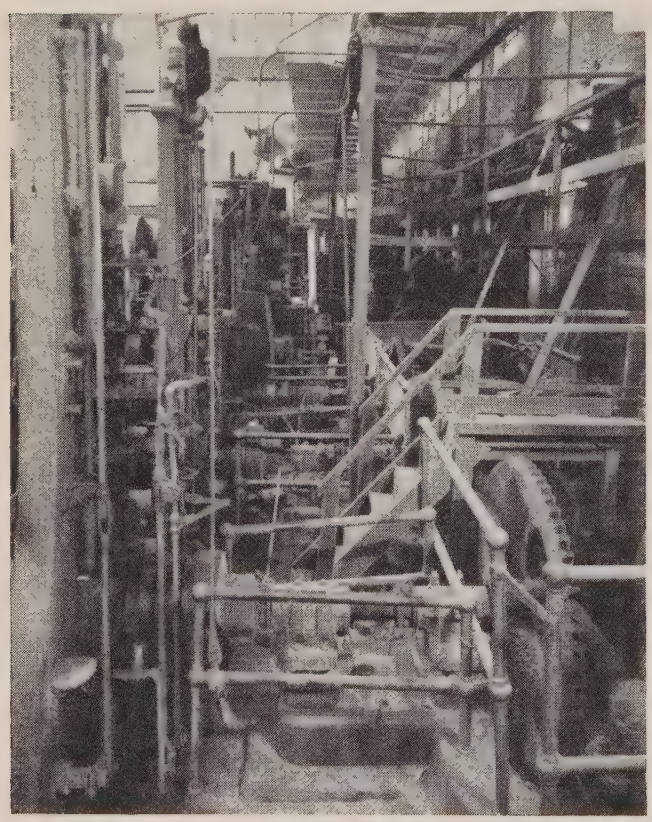

***Fig. 1. The old Marshall hookup at its worst***

As paper-machine speeds increased, this open bevel gear arrangement showed limitations. The first trouble was vibration communicated to the rolls of the machine through the in-drive shaft fitted with crude horn couplings. The frames would shake and many times affect the passage of the paper from one section to another.

The early erection of paper machines was upon two lines of wooden beams which usually became soaked in oil and sometimes turned spongy from rot. Any misalignment or badly worn wooden cogs, or both, was a serious handicap to the machine tender. The rigid cast-iron bed frame of bevel gears and pulleys helped to steady the drives, but even these anchorages were pounded out of position, or the anchor bolts were actually broken, increasing the vibration to real jolts.

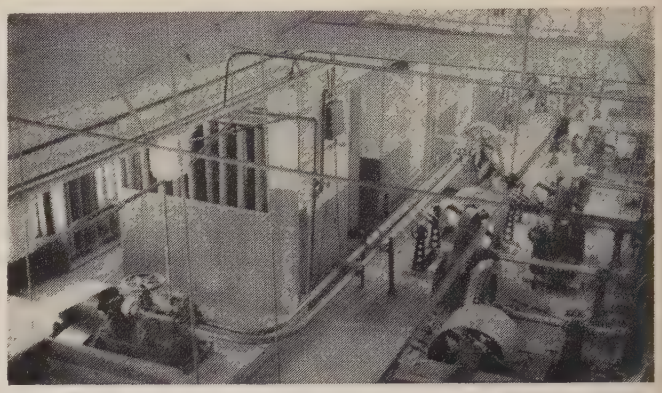

***Fig. 2. Hydraulic variable speed transmission*—(Waterbury)**

Sometimes the binding of journals and bearings would develop hot boxes or rip out the wooden cogs when started up, to the utter disgust of a papermaker.

***Enclosed Bevel Gears***

The "tearing of hair" of mill superintendents was heard by the machine builder and he did something about it. He designed the enclosed bevel gear drive running in oil.

Competition, the well-spring of the American enterprise system, called for lower costs of paper products. The engineer and the machine builder had to produce wider and faster machines. Power demands increased, which, in turn, called for a still more rugged quarter-turn drive. The enclosed bevel gear brought about the required rigidity. The anchorages of the two right angle shafts afforded stability from sudden reversals of rotation or just plain starting of a section of large mass.

During the years of development the pleadings of the papermaker developed into downright anger because of speed variations when changes of load took place. What could the engineer do about it? This phase of the problem extended beyond the machine manufacturer and into the provinces of the electrical engineer. The use of the Ward Leonard system of variable speed control had been used elsewhere on a direct-current motor. Why not apply it to a paper machine? The setup was accomplished usually by a source of power from a separate generator, usually direct-connected to a motor. The steam engine continued to be used for driving the constant speed end of the machine, and in turn, afforded the necessary exhaust steam for the driers. Naturally there were many combinations of steam engines and electric motors, too numerous to mention here.

Between 1910 and 1920 the small steam turbine had been developed and came into the picture, either to drive a direct-current generator or some mechanical variable speed hook-up to the bevel-gear drives on the various sections of the machine. There were so

many variations of the generator-turbine hook-up that the author will not take time to describe them here.

***Hydraulic Drives***

After the first World War, certain ingenious engineers thought that the so-called hydraulic drives, which had been used with great precision for turning

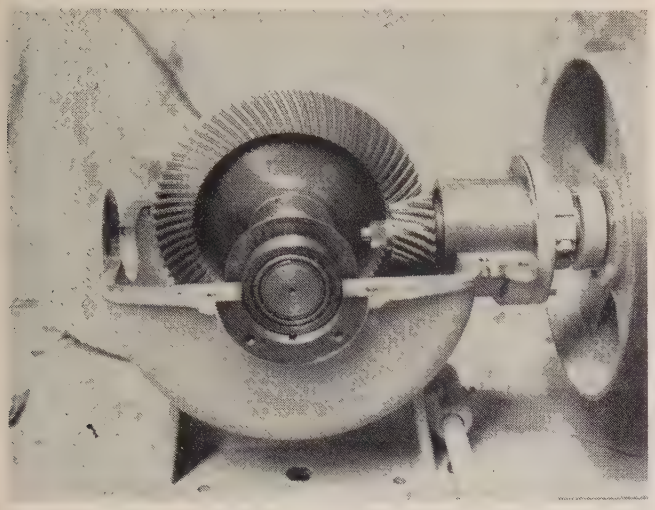

***Fig. 3. The spiral bevel gear*—(Beloit)**

gun turrets for battleships, could be used to handle the variable speed end of a paper machine as a whole. There are a few installations of sectional drives of this type where it is direct-connected to the respective rolls of the machine, and, as such, eliminated the necessity for any clutch because its speed could be dropped to zero when necessary. This equipment took up little space and was free from dust and dirt, and it afforded a smoothly regulated speed range. Briefly, it consists of a pump, or a generator end, connected by two water-jacketed pipes to the motor end, and by means of the regulation of multiple pistons, transmits variable volumes of oil to the motor which in turn responds with a variable speed. The drive is carefully designed and accurately built. Maintenance is usually handled by returning the unit to the shop where it was made by first slipping into position a rented, identical unit until return of the repaired one. The largest unit made is 300 hp. Currently it is being used in making high-grade tissues, writing, board, kraft, and parchment. A typical layout is shown in Fig. 2.

Another type of hydraulic drive involves a variable speed prime mover. This prime mover, connected to a shaft, drives a series of variable displacement hydraulic pumps. Each of these pumps is hydraulically connected to a constant displacement hydraulic motor. With this layout it is possible to change the speed of the machine as a whole by changing the speed of the prime mover. The change in draw is accomplished through remote control by altering the displacement of the individual pump driving that section. The operating efficiency is high. There are a few of these drives in operation, and, as far as can be determined, are giving entire satisfaction. The moisture around any paper machine, of which there is a great deal, has no effect on this type of drive. The internal parts are rather complicated, and, as such, are no subject for the machinist or millwright. Repairs are made the same as described above. Thus far this type of drive has been applied to relatively slow speed machines. Maximum horsepower per section is 100.

There is something about the mind of the average American that he is not satisfied with the speed at which he does anything. The author well remembers in the design work for one papermaker that a machine was built which had a capacity $2^1/_2$ times anything he had operated before. Was he satisfied with that increase in production? He was not. He loaded this machine way beyond design values until it actually broke down, just so he would know how to proceed at a slim margin of safety under breakdown point and not run the maintenance too high. So it has been with the bevel gear. It had to perform with ever-increasing tooth pressure. First, the teeth had to be cut, and in order to reduce the noise and promote smoothness of operation, cut cast-steel pinions and wooden inserts for teeth in the large gear had to be provided.

***Spiral Bevel Gears***

The next step was the enclosing of a pair of bevel gears to provide improved lubrication as well as to stop spattering of grease around the drive. Still smoother operation came from twisting the teeth slightly, and the use of the spiral bevel gear became popular. It had two bearings on the shaft for the large gear, and only one bearing for the pinion shaft (Fig. 3).

For a number of years this right angle transmission with its large gear dipping in oil, plus pressure feed of the oil to other parts, appeared to satisfy the need of engineers and papermakers. Antifriction replaced babbitted bearings. Heat-treated steel was used for the gears. But the advent of the automobile brought

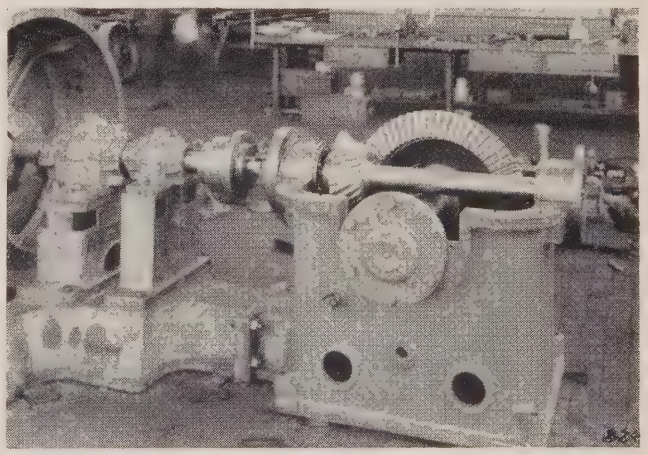

***Fig. 4. "Rosette" hypoid floor assembly*—(Pusey & Jones)**

into extensive use the hypoid gear, sometimes called the "Skew-Bevel," which afforded a more ruggedly-built setting with its two "iron box" bearings for each gear shaft. Figure 4 shows this construction.

***Hypoid Gears***

A controversy has been going on for many years

as to where the spiral bevel combination should be used versus the hypoid gear. Heavy shock loads on very large machines like those encountered in calender operation have left no question but to use the hypoid unit. On the other hand, large high-speed machines are running most satisfactorily today with spiral bevel gears. These gears have slightly higher efficiency. The author remembers one instance where

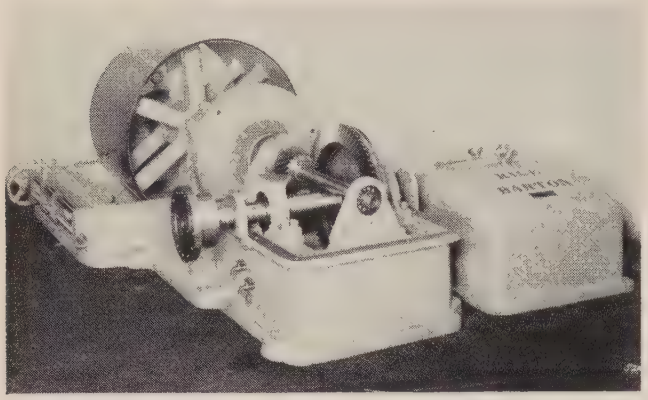

***Fig. 5. Hypoid drive with clutch*—(Rice Barton)**

he was consulted regarding the two types of gears, and recommended the spiral bevel because it was 15% cheaper and filled the needs satisfactorily. The party bought hypoid gears for the replacement of open mortise on all the drives of a small slow-speed machine, and threw "down the river" approximately $4000.

When spiral bevels handle 1000 f.p.m. and more on a 218-inch machine, it is utter folly to waste one's money on a more expensive type. On the other hand, there are some things to be said for the hypoid gear. This more rugged design affords great stability where operating conditions are unusually severe. Some paper machinery manufacturing companies concentrate on this type of drive solely, where others build both types. This dual situation will continue to exist for years to come as machine speeds mount higher and higher. Figure 5 shows a hypoid drive with clutch.

### *Flat Belts*

The two controversial drives started their respective careers with the use of flexible flat belts on cone pulleys to adjust draws anywhere from 15 to 25%. The limitations of the belt drive are now being approached in both the width of the belt and its lineal speed. Small pitched taper on long-faced cone pulleys have permitted the use of belts up to 14 inches in width, which at 5000 feet of speed will handle 200 hp. Sixteen inches is considered the maximum width of belt to use which corresponds to 230 hp. at highest belt speed.

### *V-Belts*

Now the V-belt drive has entered the picture. Originally these belts had relatively short life when operating under severe conditions. This situation led manufacturers to redesign the belts to maintain flexibility but to add greatly improved operating tensile strength and wear. Naturally such belts could not be used on paper machine drives without a reasonable amount of variable speed adjustment for respective draws. A series of adjustable driving sheaves to give varied pitch diameters, controlled from the front of the machine, afforded a compact unit which can out-transmit the flat belt in horsepower. Efficiency of transmission has been improved and a constancy of speed maintained once the adjustment of the draw has been set. No basement is required, and only a relatively narrow space from the rear of the machine to the wall is required.

Figure 6 shows a compact, easily adjusted V-belt drive snugly connected with the hypoid corner units. The speed of the line shaft is normally high, reaching to 1200 r.p.m., permitting the use of smaller line shafting and relatively high-belt speeds thus reducing the number of belts even with the transmission of large horsepowers. The whole setup is simple, of high efficiency, of low cost of installation, and in case of trouble can be easily repaired by a millwright.

Another interesting drive developed in the 1930's is shown in Fig. 7, comprised of several simple power transmission units snugly packaged into an enclosing case with the variable speed, likewise controlled by V-belts running vertically from the adjustable pitch lower pulleys, obtained by changing the center distance between the drive and the driven units. This type of drive fits into a small space behind the machine and likewise requires no basement. Similar to the previous setup, the variable speed shaft extending from unit to unit can be operated at high speed. High transmission efficiency is also obtained.

Recently there has been developed the completely modern stream-lined V-belt short center drive shown in Fig. 8. It provides a highly efficient unit interlocked through vari-drive V-belts to a high-speed

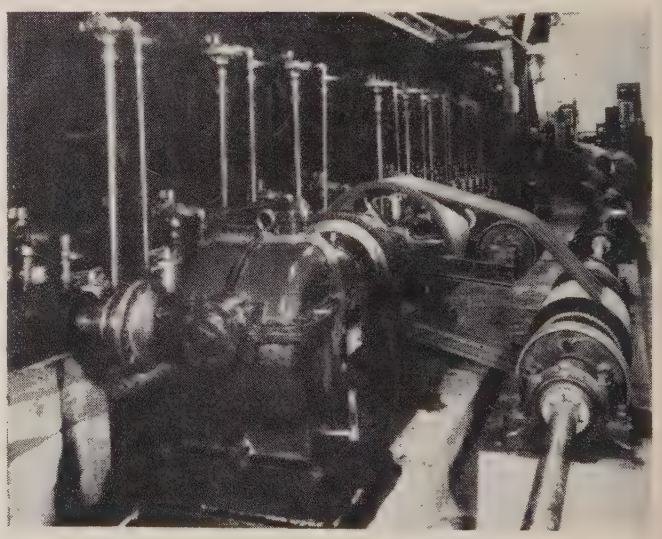

***Fig. 6. "Selective" drive*—(Sandy Hill)**

shaft running the length of the paper machine. Belt tension is regulated by a weighted pulley working with gravity. The driving shaft is placed as near the drive as the drive is from the rear of the paper machine. There are five standard units employed with flexibility of gear ratios rather than to design each set of drives specially for a new machine, thus keeping engineering and manufacturing costs at a minimum.

This is standard practice with some of the manufacturing companies. There is complete accessibility to all parts.

Inching and reversing drives are important units in a position like the calender where a plugged machine has to be cleared promptly. Figure 9 is a typical example.

***Helper Drives***

Helper drives are placed on a machine wherever slippage or undue strain is placed on a felt or wire. The positions where they may be used to advantage are on the breast roll, dandy roll, guide and wire carrying rolls, as well as cylinder molds. Top press rolls, dual press, pressure rolls on Yankee driers, and fly rolls on high-speed machines have helper units. Wherever a felt has high tension from being used as a transmission belt in turning rolls or felt driers, or both, helper motors on one or more driers can be the means of materially lengthening felt life or cause the purchase of a lighter and less costly felt. As machines go higher and higher in speed, helper motors will be more common, approaching the day when every roll or cylinder will be driven. These drives can be the means of reducing the size of a driving unit and of easing up on large flat belts on cone pulleys.

If a paper mill engineer is uncertain whether or not he should recommend the spending of money for

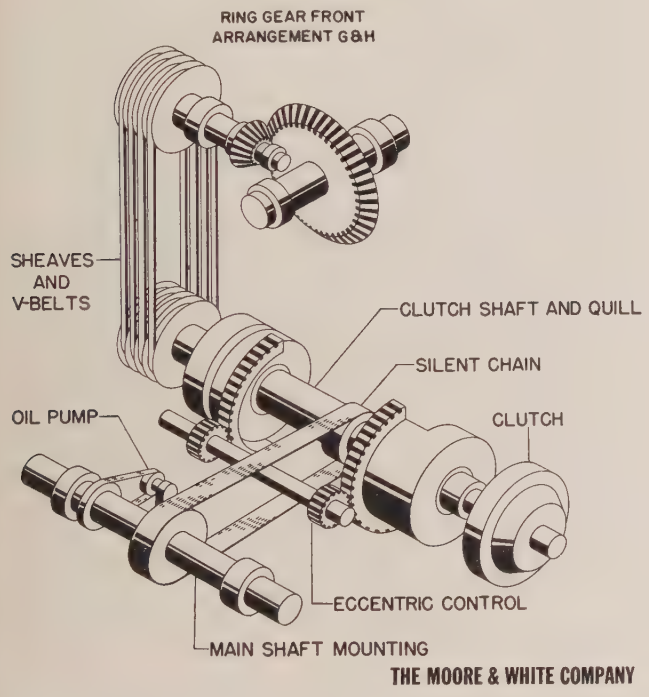


***Fig. 7. Schematic arrangement of Syco drive*—(Moore & White)**

any of these items he may elect to have them added after the machine is in operation, and so advise his superior to that effect. Anyway, he has protected himself, and the management of the company can't call him on the carpet for not having thought of it. Subsequent application is easy. The source of power for helper motors is usually a separate motor generator set equipped with a constant current regulator for the operation of the whole group once the speed of the machine has been set, or it may be run in parallel with the driving motor where there is a sectional drive.

The cylinder machine drives are similar to those on a straight fourdrinier. Generally, the power required per unit is only a fraction of that required for high-speed machines because of the relatively low speeds at which they are operated.

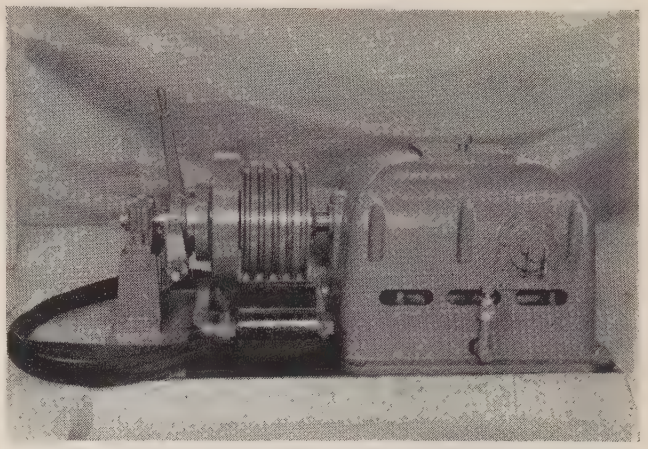

***Fig. 8. The "Simplex" spiral gear drive*—(Bagley & Sewall)**

Reels usually travel at speeds adjusted to proper paper tension to obtain the desired hardness of roll, and generally employ drives similar to those on the rest of the machine. The smaller machines and lower range of speeds often employ revolving reels with shell cores which require special variable speed motors and slip belts or are driven from the adjacent calender indrive shafts.

The worm gear is not recommended as a paper machine quarterturn drive because of relatively low efficiency and consequent heat generation.

## CLUTCHES

The clutch as a subject has not been dealt with thus far because a discussion of it can best be handled alone. It is a necessary evil, but unless the sectional electronic or hydraulic drive is used, some form of clutch must be applied. The demands of mechanical power transmission have caused engineers to improve them extensively. The application on the paper machine requires a simple design capable of smooth acceleration of massive bodies requiring nearly constant torque as well as bodies with very high-starting effort, and sometimes frequent repetition of this starting effort over a period of trouble on the machine. Great quantities of heat are thereby generated and must be dispersed without "burning up" the clutch. All kinds of clutches have been tried out on paper machines, with the consensus today that either a multiple plate exposure or peripheral plate contact to sealed pockets expanded with compressed air is the best combination. Rigid plates of steel against composition disks on ever so many machines have the disadvantage of low percentage of contacts due to distortion from heat or misalignment, or both. Always place a clutch in position, clear from all other parts of the drive.

Magnetic clutches, which grew somewhat into favor

in the 1920's, have faded into unpopularity because of the troubles had with them. The clutch picture is not settled, in spite of the strides already made to perfect them. Someone is going to develop a device which will satisfy all requirements of the paper machine without the generation of so much heat. Since variable speed shafts are turning 1000 r.p.m., the author predicts that some application of the fluid

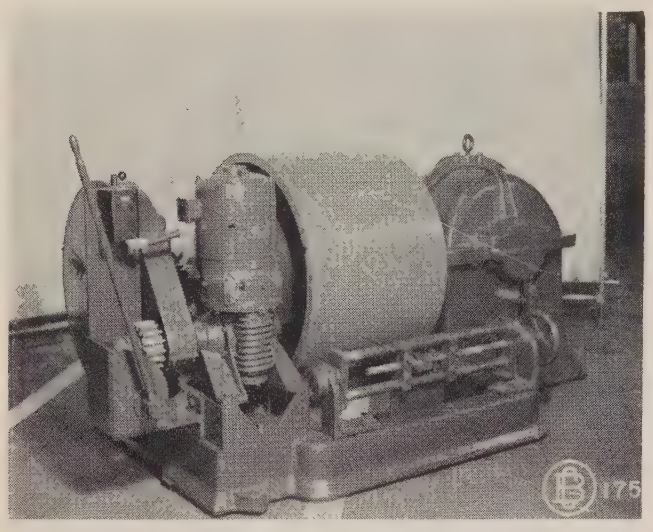

*Fig. 9. A reversing and inching drive*—(Black Clawson)

drive in conjunction with a very simple, inexpensive holding clutch, centrifugal coupling, or clamping ring, to be applied automatically at the desired speed, will be the form that this improvement will take. Its proper development should remove some of the worries of the paper mill engineer.

The horn coupling on the indrive shaft, like the clutch, can be treated alone. Its many headaches need not be given here. It was a short step from the open jaws to the refined, totally enclosed, internally lubricated gear tooth connection now in almost universal use.

The driving of the variable speed end of the paper machine has been and still is being handled by brush shifting alternating-current motors. This type of drive developed a wave of popularity in some foreign countries, with a number of installations still operating in the United States. No quarrel is picked with its application, but the ease of remote control and greater speed range of the Ward Leonard field control motor developed a more desirable application.

## SECTIONAL ELECTRIC DRIVES

Believe it or not, the first sectional electric drive was installed in 1909. Each section of the machine was driven by a slow-speed direct-current motor. Each motor, in turn, was connected through cone pulleys to a synchronous motor. These motors were locked together electrically. The variation in draw was obtained by shifting the belt on the cone pulley. A number of these drives are still in operation.

The synchronous regulator, with its small cone pulleys, soon replaced the synchronous motors and large cone pulleys resulting in a smaller and less expensive and simpler hook-up as well as a more accurate control of speed.

Speed regulator engineering has been a subject upon which electrical machinery manufacturing companies have spent a great deal of thought and experimentation. Electronic amplification appeared to be the medium through which most accurate and rapid response in speed control could be obtained.

Not until 1937 could it be said that reliable electronic tubes had been manufactured. Rapid development followed, and the first complete sectional drive with electronic regulators was installed in 1941. Inherently it is a speed regulator, and so rapid in response that one operator facetiously stated that it began to regulate even before a section of the machine started to slow up.

Nothing galls a papermaker more than an unwanted speed change on a paper machine or a jordan. The uncanny electronic speed regulator will control the speed of a section of paper machine to within plus or minus 0.2 of 1%, if everything is properly adjusted. It also shows immediately any change in the draw. Another advantage is the control and elimination of hunting at high speeds.

Until recent years the single generator system has been employed. After World War II the multiple generator system, each section motor having its own separate generator, was developed and has been installed on a large number of machines. One or all sections can be started simultaneously. Each motor having its own generator means constant speed unaffected by load changes on any other part of the machine. This is shown diagrammatically in Fig. 10. This type of drive, looking from the dry end of a machine, is shown in Fig. 11. A close-up of a single drive is shown in Fig. 12.

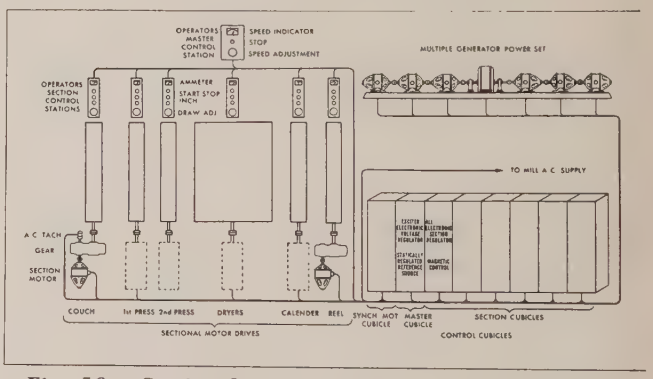


*Fig. 10. Sectional motor diagram*—(Westinghouse)

The mechanical simplification of the layout is an answer to the paper mill engineer's prayer. On the other hand there is a greater complication of electric circuits which may be a greater potential source of trouble.

The controls for sectional drives are varied in character and too involved to describe in detail here. The several motors have controls to keep them in step as well as other controls in connection with draw and tension. Many of these drives, operating very satisfactorily, do not have electronic equipment, and as such, may be purchased at a somewhat lower price.

As mentioned before, the amplified electronic sectional drives are relatively new, but in spite of this fact, there are a large number in operation and others being erected at the present time. Some paper mill engineers feel that the control equipment and connections are too complicated and the most urgent goal should be simplification. A defect, let us say, in a resistor used in a Ward Leonard system is not too difficult to locate, but a hundred more places where trouble can arise in the electronic control equipment, many times takes an extended period of time to locate and remedy a trouble.

The preventative maintenance urge has not hit the manufacturers as it has the electrical paper mill engineers. A major study, if not already started, should begin at once to devise two series of procedures; (1) to improve and simplify the technique of testing for future trouble, and (2) to point out and provide the necessary facilities to quickly diagnose trouble when it occurs in order to get the machine into production again. As one manufacturer said, the electronic sectional drive does have its growing pains. So does anything that is newly created; it is the price of perfection. Further study should be made in lightning protection.

*Fig. 11. Sectional electric drives*—(General Electric)

This may look like the dark side of the picture, but it is not meant as such because the author firmly believes that so long as paper is made on a fourdrinier machine the call for speed will be forever upward; and the sectional drive has what it takes. If someone had said 20 years ago that a paper machine would be operated at 2600 f.p.m., he would have been called a visionary. The author predicts that they will be running 3500 f.p.m. in 1969. That range may extend beyond the scope of mechanical development used in the present corner drives.

The author would be remiss without mentioning that the electronic drive which the average reader connects with high speeds, has been installed successfully on relatively slow-speed machines where great precision of speed control is necessary.

## MACHINE SPEEDS

The speed at which a paper machine runs is an interesting as well as an important economic factor in the successful operation of a paper mill. The author has talked with many mill operators on this subject. The spread of speed is large, depending a great deal on the equipment used as well as the specifications of the paper made. His study of this subject has led him into some interesting channels of thought. For example, what does the over-all picture of the manufacture of paper groups look like when translated into graphs? While the speed of the machine is not a criterion in the selection of a type of drive, nevertheless, it is revealing to see the relative speeds at which different types of drives have been employed in current practice.

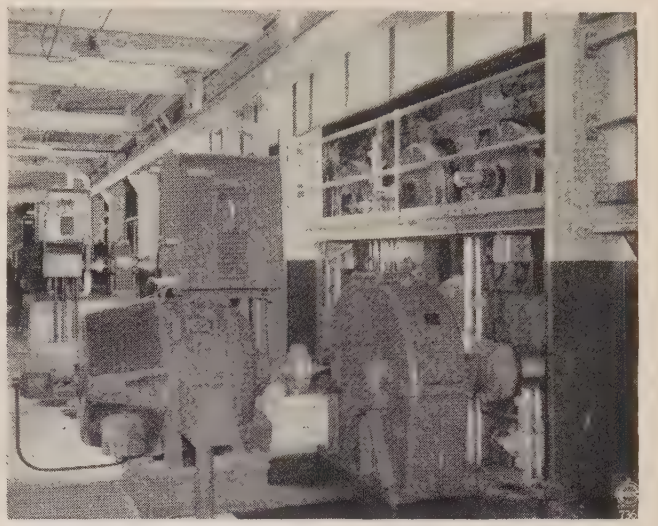

*Fig. 12. Close-up of a sectional drive*—(Reliance)

Figure 13 shows in full lines the spread of present machine speeds. Broken lines at the upper end of these graphs indicate the areas into which more daring souls have ventured and perhaps at times to return to full line region when stock or other conditions are abnormal. The upper ends of broken lines also include design areas. Two graphs are worthy of comment. Strange as it may seem, facial tissue is manufactured anywhere from 350 to 2600 f.p.m. The lower end of this graph refers to a cylinder machine and the upper end to a Yankee. Newsprint is made entirely on the straight fourdrinier. One wonders at the economic status of companies at the lower ends of these two graphs. Since these graphs, in general, cover groups of paper manufacturing (there are approximately 100 different kinds of paper and board made today, which number has no reference to color or furnish), it is obvious that a particular grade of paper which falls at the lower end of a graph may still be considered as being made at the top speed. Weight

of sheet is of course an important element of speed range within a group.

If you find one of your paper machines operating at the lower end of any graph, question yourself as to why you are there. There may be good and sufficient reasons, however, an examination of the situation may result in an improvement of your speed.

## HORSEPOWER REQUIREMENTS

The horsepower requirements of drives on different sections of a paper machine is a subject of great importance. Some years ago several of the electrical manufacturing companies worked together toward the common end of developing a table of factors to be set against the different kinds or groups of papers manufactured on both the cylinder and fourdrinier. These factors, multiplied against the number of inches of width of the machine and the speed of the machine in hundreds of feet per minute, gives a theoretical horsepower required to operate a given section of a machine. Varying safety factors were added to the results of these calculations, thus giving the rated horsepower for each section. Curves can be found on TAPPI Data Sheets 145A, B, C, D, and E, entitled, "Power Requirements for Paper Machines."

Another interesting study has been the capital cost per horsepower of the various types of drives now installed. Figure 14 gives the approximate values in shaded areas. These graphs are general in character

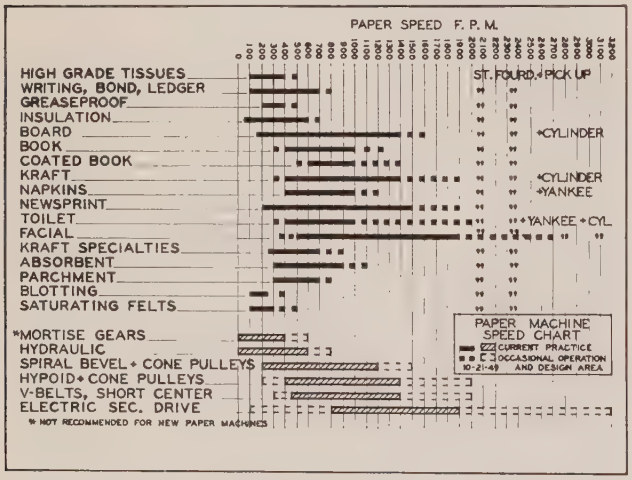


***Fig. 13. Paper machine speeds for various grades of paper and ranges for various types of gears***

since accurate figures naturally depend on such factors as the size of the machine, as well as the number of sections and the speed at which the machine will travel. However, there are interesting comparisons. The open mortise gear drive naturally was a low cost arrangement. The hydraulic drives went to the other extreme. The packaged V-belt drive, though limited in capacity, dropped back in cost to become competitive with drives shown above. The development of the V-belt short center drives brought out an economical design competitive with any of the graphs shown above it. It will be noted that two bevel gear graphs broaden out in the shaded areas because of variations in design and double reductions in speed. The spiral bevel drive averages slightly lower in cost than the hypoid. The sectional electric drive, thought by many to be more expensive in first cost, is placed in a most competitive position. Naturally one type of drive may be less expensive than another, but the total cost to

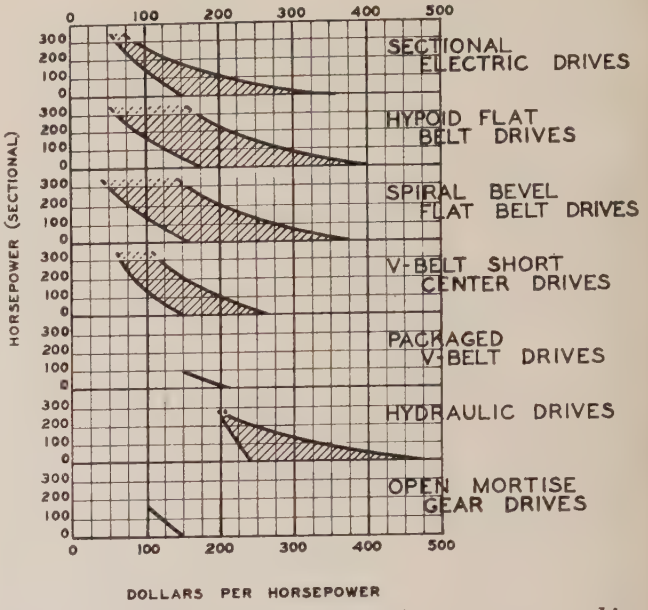


***Fig. 14. Horsepower cost comparisons paper machine drives, including prime mover and up to paper machine. (Equipment not installed)***

the point of operation may be a different picture. Unfortunately time did not permit the completion of this phase of the project.

## SUMMARY

The author has tried to give a brief historical background in which there has been developed the different types of drives on paper machines, together with a general description and illustrations of their construction. Graphs have been developed from the large amount of data collected which can be a guide to a paper mill engineer in his study of the drive required for a particular situation. At a glance he may ascertain a rough relationship of costs of the various drives before purchase.

The use of facts or opinions brought forth in this paper should be made only after an evaluation of the various factors surrounding a proposed paper machine. They are, the speed to be run with due allowance for future growth, as well as allowable variation of this speed; the mass or inertia of the respective sections together with desired rates of acceleration and deceleration; the amount of sequential draws and limits thereof; and finally, appraisal of reversing and inching operations and available floor space. All controls should be at most convenient positions.

Engineering knowledge is built upon experience and the foundation upon which we build engineering intelligence. It is engineering wisdom only if the test of time proves a machine to be not wanting. That means under good management, both the purchaser and manufacturer will benefit.

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# The Precision of Viscosity Measurements on Solutions of Wood Pulp in Cupriethylenediamine

J. D. McLEAN and FORESTIER WALKER

**The variables affecting the precision of the cupriethylenediamine viscosity test have been investigated, and the probable sources of error in the test evaluated.**

DURING the last few years an increasing number of pulp mills, in which viscosity control is important, have adopted the cupriethylenediamine method for the determination of cellulose viscosity. The growing popularity of this test is evidenced by its adoption by TAPPI as a suggested method. In many cases the cupriethylenediamine method has replaced the older cuprammonium method. Numerous advantages are claimed for cupriethylenediamine over cuprammonium for cellulose viscosity measurements (*1*, *2*, *4*). The chief advantages are: (1) ease of preparation of solution, (2) stability of solution, and (3) decreased effect of oxygen on the sample and solution during the dispersion.

The cupriethylenediamine ball-fall method has been used in both the Fraser Co. research and control laboratories for over three years. Of the methods used to date it has been found to be the most satisfactory although on occasion difficulty has been experienced in obtaining the desired precision in the control of pulp bleaching. Because of these variations an investigation was recently undertaken to determine the variables in this method and to measure their effect on the precision of the cupriethylenediamine viscosity test.

## PROCEDURE

The preparation of solutions and sample and the procedure for carrying out the test were similar to that of Hatch (*1*, *2*). The ethylenediamine to copper ratio of the solutions was carefully adjusted to 2.00, except in those cases where studies of this ratio were made. The concentration of ethylenediamine was determined by titration, using methyl orange indicator, and after titration the end point was checked with a glass electrode. The pH at this end point was 3.3 to 3.5 (*1*). All measurements were made using the falling ball method with a $1^1/_2\%$ cellulose concentration and a ball-fall time of approximately 20 seconds.

A standard procedure was used throughout this investigation, and deviations from it were made only to study the effect of the different variables; such deviations are recorded with the experimental data. The experimental technique was that of Hatch (*2*) with only minor variations. The details of this standard procedure are as follows:

A sample of dissolving grade pulp weighing approximately 0.9 gram is placed in a Waring Blendor, about 500 ml. of water added, and the pulp allowed to soak for a few minutes. Then the Blendor is run at full speed for 30 seconds, and the pulp suspension filtered on nainsook cloth in a 9-cm. Buchner funnel. The pulp is carefully washed with two 25-ml. portions of redistilled acetone in such a way that the formation of the mat is not disturbed. The mat is then quartered and placed in a Moisture Teller for 3 minutes at 190°F. On removal from the Moisture Teller the sample is quickly placed in a small aluminum weighing can so that two opposite quarters are uppermost. The two top sections of pulp which are to be used for the viscosity test are placed in a 2-ounce medicine bottle. The remainder is tested for moisture content in the usual way. The value for moisture thus obtained is used in calculating the weight of pulp taken for the test.

The air in the bottle containing the sample is displaced with a stream of nitrogen. The required amount of 0.167 *M* cupriethylenediamine solution, prepared according to the method of Hatch (*1*, *2*) is added, followed by the 1.00 *M* solution. More nitrogen is applied and the cap placed on the bottle. The samples are shaken in a mechanical shaker for exactly 3 minutes, and the solution poured into viscosity tubes which are placed in a constant temperature bath at 25.0 ± 0.05°C. At least 15 minutes is allowed for the solution to reach equilibrium, then the standard $^1/_{16}$-inch aluminum balls are dropped through a 15 cm. depth of solution and the viscosity determined. In cases where bubbles form at the surface of the solution in the tubes and interfere with dropping the balls, such bubbles are removed with a glass rod.

## EFFECT OF VARIABLES

### *Ratio*

The ratio of ethylenediamine to copper is critical. In this investigation it has been found that there is a change in viscosity over a wide range of ethylenediamine copper ratio as shown in Table I. Ratios covering the range 1.9 to 2.1 were rechecked with a different pulp and solution, Table Ib. No mention has been made in the literature regarding the correct ratio for this test other than to specify that it should be less than a given value. A maximum value of 2.00:1 is specified by TAPPI (*6*), Hatch (*1*, *2*), and Straus and Levy (*5*). The latter report that at a ratio of 2.25:1 the solution has no solvent power and that if ethylenediamine is added to a solution of cellulose to bring the ratio up to 2.25, cellulose will precipitate out of solution. In this work a $1^1/_2\%$ cellulose solution with a ratio as high as 3.12:1 has been prepared.

Straus and Levy (*5*) report that a solution in equilibrium with excess cupric hydroxide has a molar ratio of 2:1. They refer to a paper by L. J. Jolley (*J. Textile Inst.* 30: 122–141, 1939) in which he reports an equilib-

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**Table Ia. Variation of Viscosity with Ratio, Range 1.7 to 3.1**

| Ratio | Viscosity, cp. Average | Viscosity, cp. Standard deviation |
|---|---|---|
| 1.73 | 203 | 3.6 |
| 1.90 | 212 | 6.6 |
| 1.99 | 213 | 4.1 |
| 2.00 | 214 | 3.0 |
| 2.06 | 225 | 4.9 |
| 2.18 | 231 | 3.9 |
| 2.20 | 232 | 6.8 |
| 2.47 | 253 | 4.3 |
| 2.57 | 269 | 4.6 |
| 2.85 | 278 | 5.6 |
| 3.12 | 296 | 4.0 |

**Table Ib. Variation of Viscosity with Ratio, Range 1.9 to 2.1**

| Ratio | Viscosity, cp. Average | Viscosity, cp. Standard deviation |
|---|---|---|
| 1.91 | 191 | 3.3 |
| 1.92 | 193 | 1.2 |
| 1.93 | 199 | 3.4 |
| 1.96 | 189 | 0.6 |
| 1.97 | 195 | 2.9 |
| 1.98 | 201 | 1.4 |
| 1.99 | 196 | 2.7 |
| 2.00 | 202 | 0.9 |
| 2.02 | 203 | 1.8 |
| 2.03 | 203 | 1.6 |
| 2.06 | 205 | 2.7 |
| 2.11 | 207 | 1.3 |

rium ratio of 1.78 to 1.89, and to a paper by W. Traube (*Ber.* 44: 3319–3324, 1911) who reports a ratio of exactly 2:1 independent of dilution. In this investigation it has been found that the equilibrium ratio is proportional to the concentration of ethylenediamine. In general, when following the standard procedure the equilibrium ratio has been approximately 1.90 to 1.95:1, but when the concentration of ethylenediamine has been low a ratio of 1.73:1 has been obtained and when a minimum of water was present resulting in a high concentration of ethylenediamine, a ratio of 2.16:1 resulted. The wide divergence in the published data can be explained by this effect of ethylenediamine concentration.

The results of this investigation, as given in Table I, are in agreement with Hatch (*1*) who reports viscosity results 3 to 5% high at a ratio of 2.1:1 and 25 to 30% high at a ratio of 3:1, although they do not agree at lower ratios.

The data as given in Tables Ia and Ib appear somewhat inconsistent since a small change in ratio is not always followed by a corresponding change in viscosity; however, the determination of ratio is not precise in the third significant figure. If the results in these tables are graphed on a scale that allows for the precision of the ratio determination and the precision of the pulp viscosity measurement, almost all points will fall within the plot.

**Table IIa. Variation with Time in Waring Blendor**

(Pulps A and B were samples taken from different sheets of the same bale)

| Time of dispersion, (sec.) | Viscosity, cp. Pulp A Average | Pulp A Standard deviation | Pulp B Average | Pulp B Standard deviation |
|---|---|---|---|---|
| 15 | 209 | 4.8 | 209 | 1.6 |
| 30 | 211 | 3.6 | 214 | 3.0 |
| 60 | ... | ... | 211 | 5.8 |
| 300 | 206 | 2.3 | 213 | 3.8 |

**Table IIb. Variation with Time in Knapp Monarch Liquidizer**

| Time of dispersion (sec.) | Viscosity, cp. pulp C Average | Viscosity, cp. pulp C Standard deviation |
|---|---|---|
| 15 | 258 | 3.0 |
| 60 | 253 | 0. |
| 300 | 245 | 5.0 |

***Dispersion***

The data in Table II show that the viscosity is not appreciably affected by dispersion time in the Waring Blendor, although this is not the case in a Knapp Monarch Liquidizer.

**Table III. Variation of Viscosity with Pulp Drying Temperature**

(Drying time 10 minutes in Moisture Teller)

| Temperature, °F. | Viscosity, cp. Average | Viscosity, cp. Standard deviation | Viscosity, cp. Average | Viscosity, cp. Standard deviation |
|---|---|---|---|---|
| 165 | 209 | 7.6 | 207 | 8.0 |
| 200 | ... | ... | 210 | 4.9 |
| 235 | 200 | 2.9 | 210 | 3.7 |

***Drying Temperature***

An increase in the drying temperature of the sample in the Moisture Teller affects the pulp viscosity only slightly as shown in Table III. In this series of tests the drying time was 10 minutes while in the standard procedure it is 3 minutes. Under normal conditions of drying a fluctuation in temperature of a few degrees would not be expected to cause appreciable change in the viscosity of the pulp under examination. Hatch (*3*) has reported that drying pulp samples at temperatures above 200°F. will give low results.

***Solution***

The time during which the pulp is shaken during solution is critical. Samples were shaken for various lengths of time and the results, as given in Table IV, show that the viscosity is lowered with increased time of shaking. Traces of oxygen in the solution bottle will cause degradation of the cellulose, and prolonged shaking will hasten this effect. Passage of the nitrogen through alkaline pyrogallol solution did not prevent the degrading effects of prolonged shaking, but the final viscosities were higher, No. 3, Table IV. Hatch (*2*) has reported that there is no change in viscosity with time of shaking when commercial nitrogen is used. Quite possibly he worked with a very pure grade of nitrogen.

**Table IV. Variation of Viscosity with Shaking Time**

| Shaking time, min. | Viscosity, cp. (1) Average | (1) Standard deviation | (2) Average | (2) Standard deviation | (3) Average | (3) Standard deviation |
|---|---|---|---|---|---|---|
| 1/2 | 229 | ... | ... | ... | ... | ... |
| 1 | 230 | 2.4 | 218 | 2.6 | 221 | 3.1 |
| 1 1/2 | 224 | ... | ... | ... | ... | ... |
| 2 | 229 | ... | ... | ... | ... | ... |
| 3 | ... | ... | 211 | 3.6 | 216 | 2.3 |
| 4 | 232 | ... | ... | ... | ... | ... |
| 6 | 217 | ... | ... | ... | ... | ... |
| 10 | 215 | ... | 204 | 1.7 | 211 | 2.2 |

### *Oxygen*

The effect of oxygen was briefly investigated; this did not include minute amounts of oxygen which are reported to cause slight degradation, but only quantities of the magnitude encountered in routine testing. The results appear in Table V. Hatch has also reported (*2*) a low result if air is present in the bottle during solution of the sample.

**Table V. Effect of Oxygen on Viscosity**

| *Conditions during dispersion* | *Viscosity, cp.* | | | |
|---|---|---|---|---|
| | *Average* | *Standard deviation* | *Average* | *Standard deviation* |
| Gas phase, air | 200 | 0.6 | ... | ... |
| Gas phase, commercial nitrogen | 218 | ... | 211 | 3.6 |
| Gas phase, nitrogen passed through alkaline pyrogallol | 221 | ... | 216 | 2.3 |
| Gas phase air. Bottle shaken and stored 18 hours before testing | 166 | 4.8 | ... | ... |
| Gas phase nitrogen. Bottle shaken and stored 18 hours before testing | 207 | 12.0 | ... | ... |

### *Other Variables*

Apart from the normal errors which may occur during measurement of solutions and in calibration of equipment, there is one other possible source of error: equilibrium is reached slowly in a freshly prepared solution and reproducible ball-fall times are obtained only after a 10 to 15 minute interval.

### *Reproducibility of Results*

The data obtained in this laboratory indicate a standard deviation of 2.8 over a range of viscosities from 50 to 275 cp.; this agrees with previous publications (*4*, *5*, *6*).

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# Wettability of Paper Surfaces by Water and the Surface Absorption of Water by Paper

GEORGE D. KNIGHT

**An apparatus which will reveal irregularities in the surface wettability of a sheet of paper was constructed, and a technique for its operation was devised. Surface patterns produced on several papers were visually compared and the quantities and depths of penetration of dye solutions in the surface layers were calculated by means of an extension of the Kubelka-Munk relationships. The results did not correlate with standard physical paper tests. The surface wettability of paper was found to be extremely sensitive to practically any type of treatment. Results are of value in tub sizing, coating, gumming, or other converting operations concerned with the application of aqueous materials to paper, including printing with aqueous inks. The procedures may also be found to extend to solvent wettability of paper and, hence, to the fields of oil ink printing, lacquering, varnishing, and practically any operation dealing with application of fluids to paper.**

A VAST AMOUNT of work has been done on the susceptibility of paper to wetting by various liquids, but practically all the tests involve the characteristics of the whole sheet. Rarely do any of the tests take into account the time required for the initial wetting of the surface. Numerous references (*1–3*) are found which discuss the nonuniformity of wetting of paper but most authors seem to consider the irregularity as part of the whole sheet structure rather than as a function of surface characteristics.

Uneven drying and mottling and irregular absorption of inks, coating colors, etc. are real and troublesome factors. Calendering, sizing, ink, sheet formation, extraneous chemicals, refining, drying, moisture content, fillers, and type of fiber have all been blamed, singly and collectively, for the difficulties but, as a rule, the structure and composition of the whole sheet have been considered. Studies of the surface of the sheet only and of the phenomena occurring in the very small interval between the time the liquid is placed on the sheet and the time it actually wets the surface have been almost completely neglected.

There seem to be two reasons for this neglect. One is that the great importance of surface properties has never been fully appreciated. The magnitude of effects produced by minute quantities of materials is not generally known, particularly in the field of paper. For example, the thickness of contamination required to influence the property of a surface to be wet by water is about 10 A units (*4*) and layers of grease not more than 1 molecule thick can materially increase the contact angle (*5*).

A second reason for neglecting the spontaneous wetting properties of paper surfaces has been the lack of suitable means for the study of these properties. Recently, Rowland, Fronmuller, and Van den Akker described a device known as a surface wettability tester (*6*). The apparatus operates on the principle of spreading a large excess of colored fluid over a paper surface and, a very short time later (about 0.01 second), removing by vacuum all fluid which has not

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been sorbed by the paper. Patterns obtained on various papers varied from one which was smoothly colored to one which was extremely mottled with colored areas of different size and different depths of coloring.

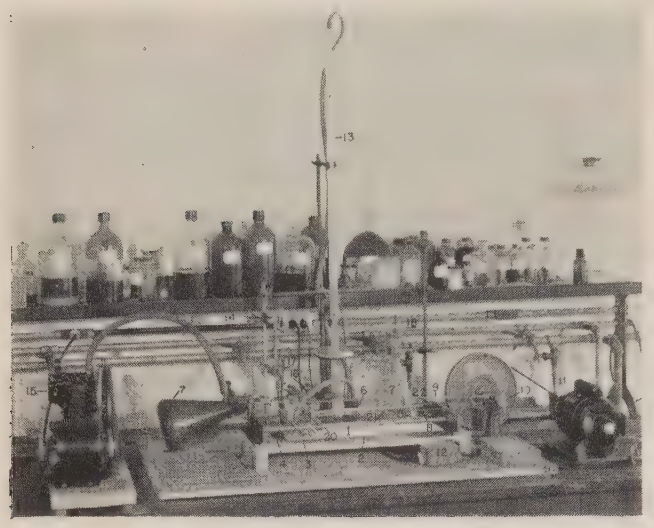


*Fig. 1. Surface wettability tester*

Key to Figure: 1. paper to be tested ($5^1/_2$ by 20 inches), 2. backing material (in this illustration, plate glass), 3. ink applicator (see Fig. 2), 4. clip to hold paper and backing material, 5. guide rails ($^1/_2$-inch diameter steel rods), 6. excess ink trap, 7. vacuum reservoirs, 8. driving cord ($^1/_8$-inch woven cotton clothesline), 9. rubber tubing to maintain tension in driving cord, 10. driving pulley ($1^1/_8$-inch diameter V-belt pulley), 11. driving motor ($^1/_4$ h.p., 1725 r.p.m. capacitor), 12. driving motor switch, 13. mercury manometer, 14. needle valve to regulate vacuum, 15. vacuum pump assembly, 16, 17, 18. stopcocks to control vacuum, 19. mounting rails for motor ($1^1/_4$ by $1^1/_4$-inch angle iron). These rails extended under the whole assembly and served to stiffen the plywood base, 20. applicator carriage (see Fig. 2), 21. driving cord release, and 22. rubber bumpers.

Using the principle of the above device as a starting point, the purpose of this work was to devise an improved apparatus for studying spontaneous wettability and absorption, to develop a procedure for operating the device, and to develop a technique for quantitatively evaluating the treated paper in such a way as to obviate subjective judgments.

## APPARATUS

Some preliminary work was done with the tester of Rowland, et al. (*6*) and several new designs of wettability testers were considered. The final design of the apparatus used in this work is shown in Figs. 1 and 2.

The holes through which the assembly bolts (23) passed in the front blade (25) were elongated so that the blade could be adjusted vertically and independently of the rear blade. The assembly of both blades could be adjusted vertically from the paper surface by placing shims at the blade connection to the skids (33). With no shims, the bottoms of the rear blade and of the skids were in the same plane. The blade clearances were measured on a surface plate with feeler gages.

Desired speeds were obtained by changing the large V-belt pulleys in the driving mechanism. The actual speed obtained was determined by measuring the revolutions per minute of the driving shaft.

The degree of vacuum was measured with the mercury manometer (13) and recorded as the difference between atmospheric pressure and the pressure in the system. Any desired vacuum from zero to the maximum could be obtained by manipulation of the four valves (14–18).

The desired backing material (about 6 inches wide and 20 inches long) was placed in position at 2 in Fig. 1.

## OPERATING PROCEDURE

With the paper specimen in place on the backing material and the applicator in starting position as shown in Fig. 1, the vacuum valves were adjusted until the desired vacuum was obtained; then 1 to 2 ml. of ink were drawn out in a line (about $^1/_4$-inch wide and $3^1/_2$-inches long) just ahead of the front blade of the applicator. (If the ink was placed in contact with the front blade, the vacuum, in most cases, would immediately draw most of the ink off the paper.) The driving motor (11) was started immediately. As the applicator was drawn along the sheet at constant speed, the front blade pushed the pool of ink ahead of it and spread a layer of ink over the paper. The vacuum slot following just behind the front blade removed any free liquid which had not entered the paper surface. The ink remaining in the paper after passage of the carriage was that which wet and entered the

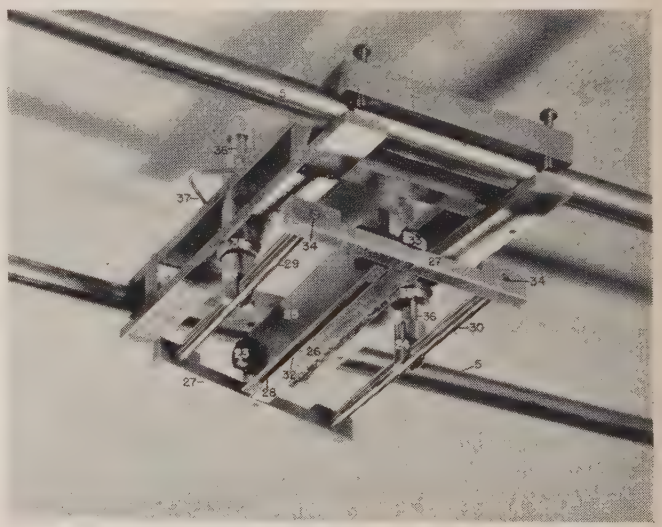


*Fig. 2. Assembly of ink applicator and applicator carriage (bottom view)*

Key to Figure: 5. guide rails ($^1/_2$-inch diameter steel rods), 23. assembly bolts, 24. mounting connections to applicator carriage, 25. front blade ($^3/_{32}$-inch thick), 26. rear blade ($^3/_8$-inch thick), 27. skids (toed-in an angle of 5°, $^1/_4$ by $3^3/_4$-inch rubbing surface), 28. vacuum slot ($^1/_8$ by $3^1/_4$-inch opening), 29. front drive bar ($^1/_4$-inch diameter), 30. rear drive bar ($^1/_4$-inch diameter), 32. undercut on rear blade (makes "effective" width of rear blade only $^1/_{16}$-inch), 33. blade connection to skids, 34. skid bearings, 35. sleeve bearings, 36. connecting rods, and 37. connection to driving cord.

paper surface during the short interval of time between spreading the ink and vacuum removal. When the applicator struck the rubber bumpers (22), the motor was stopped and the test paper was removed for examination.

## RESULTS AND DISCUSSION

### *Effects of Operating Variables*

Such variables as ink, clearance of applicator blades, speed, backing material, and vacuum were found to be important.

The effect of ink was not extensively investigated. Practically all work was done with a solution of dye in distilled water. It seemed that this type of ink would most graphically illustrate surface irregularities of importance to coating, gumming, laminating, printing with water-base inks, etc. The results with different solvents would not be comparable. For instance, areas of a paper surface which are readily wet by oil would not be wet by water.

The "ink" arbitrarily adopted as a standard was 0.5 gram of Methylene blue ZX in 100 ml. of distilled water.

The clearance of the applicator blades from the paper surface proved to be critical and adjustments were extremely difficult to make. Any selection of proper clearances, however, proved to be arbitrary. When the clearance of both blades was zero, the apparatus was extremely sensitive to minute thickness variations in a hard backing material. Large dust particles and wax pencil marks on the plate glass backing would cause dark lines on the paper surface across the direction of travel of the applicator. Apparently, when the applicator blade struck a suddenly raised portion of the sheet, it momentarily changed speed enough to leave an irregular ink deposit on the surface.

When the front blade was raised more than about 0.003 inch above the paper surface, the vacuum drew the ink away from the paper before the run was completed.

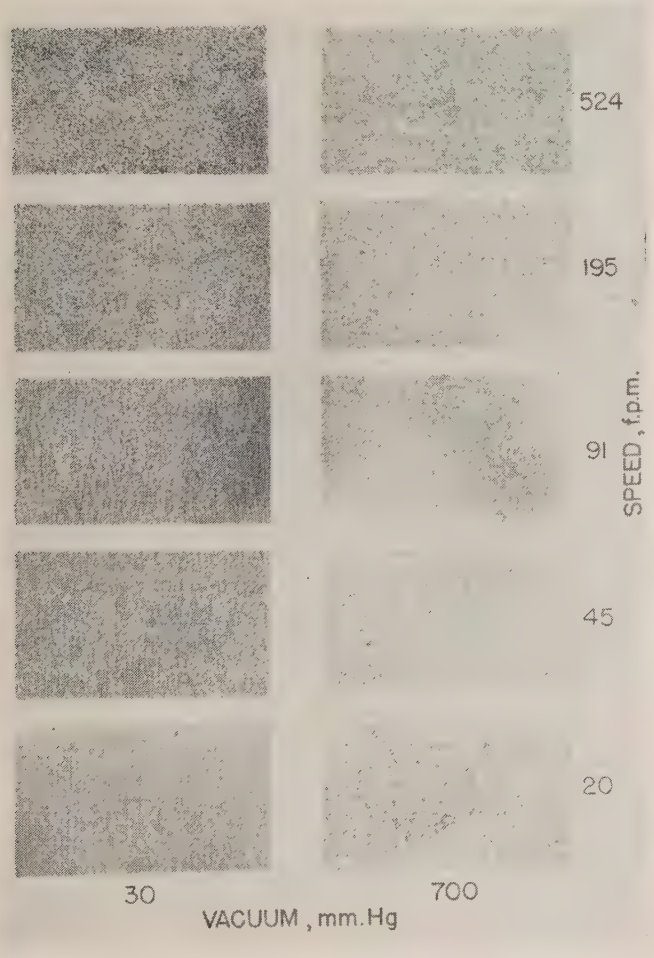



*Fig. 3. Effect of speed of ink application. Sample 11. Backing—plate glass*

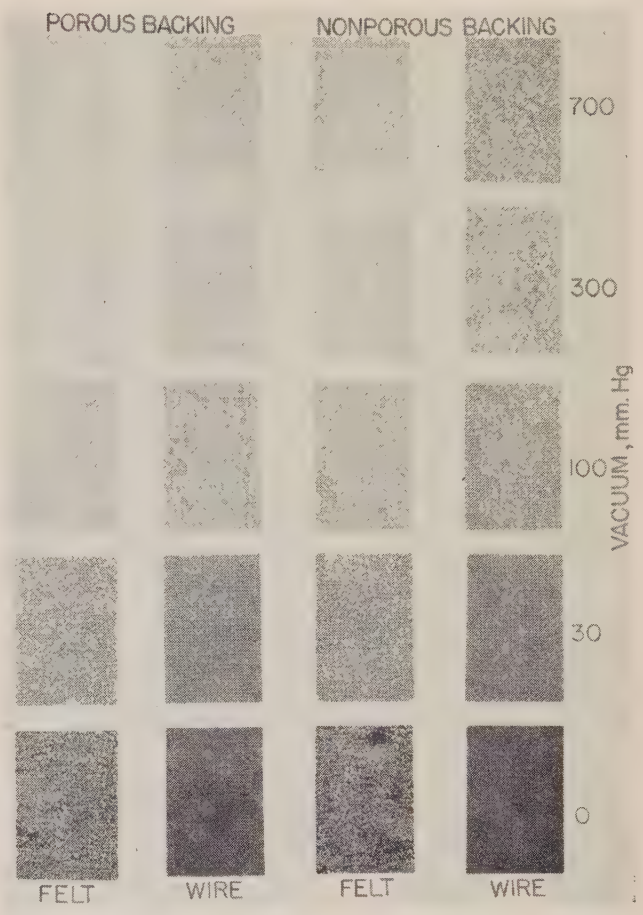



*Fig. 4. Effect of vacuum. Sample 11. Speed—91 f.p.m.*

The clearances which were selected as optimum for this investigation were:

| | |
|---|---|
| Front blade | 0.0015 inch |
| Rear blade | 0.0030 inch |

The effect of speed of application of the ink is shown in Fig. 3. As the speed was increased (the vacuum remaining constant at 30 mm. Hg), the pattern became more and more deeply colored and mottled. At slow speed, the paper surface is exposed to the ink a relatively long time and, therefore, is fairly uniformly wet. However, at 20 feet per minute, the "exposure" is only about 0.1 second before the vacuum removes the excess ink, thus preventing any large ink absorption by the surface. At higher speeds, the exposure time is proportionately reduced to intervals which are too short to permit uniform wetting. The vacuum also has less time in which to remove the excess; therefore, more ink is left on the surface.

The action of the applicator when a high vacuum is used is decidedly different from that discussed above (see Fig. 3). The chief cause of the difference is the impregnating effect described below.

The effect of the backing material can be seen in Fig. 4. At low vacuum, the backing has no noticeable effect. At a vacuum greater than about 30 mm. Hg, however, the influence of the backing becomes progressively more pronounced. The tabs backed with a nonporous material are appreciably darker colored than those backed with a porous material.

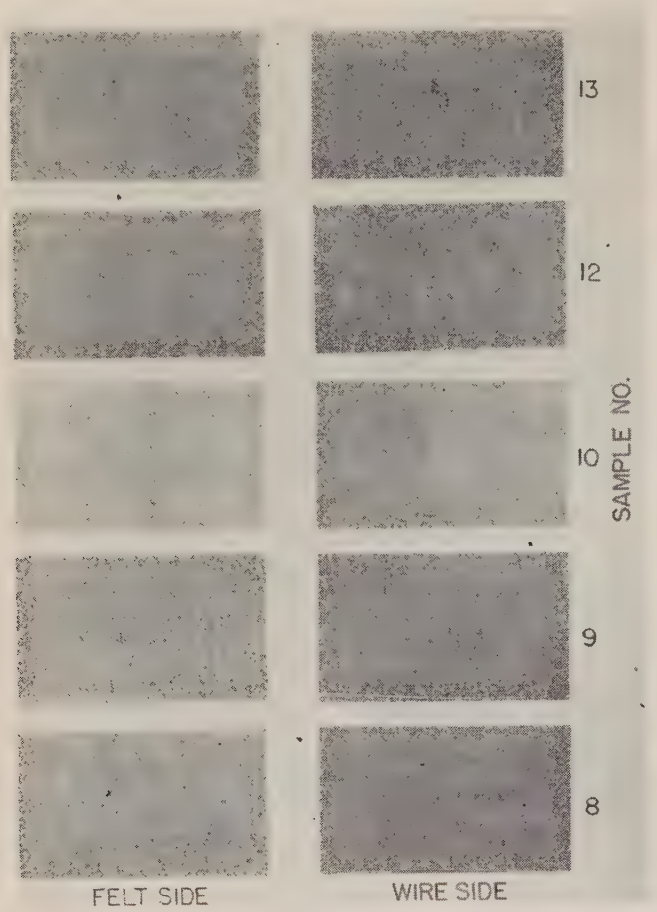
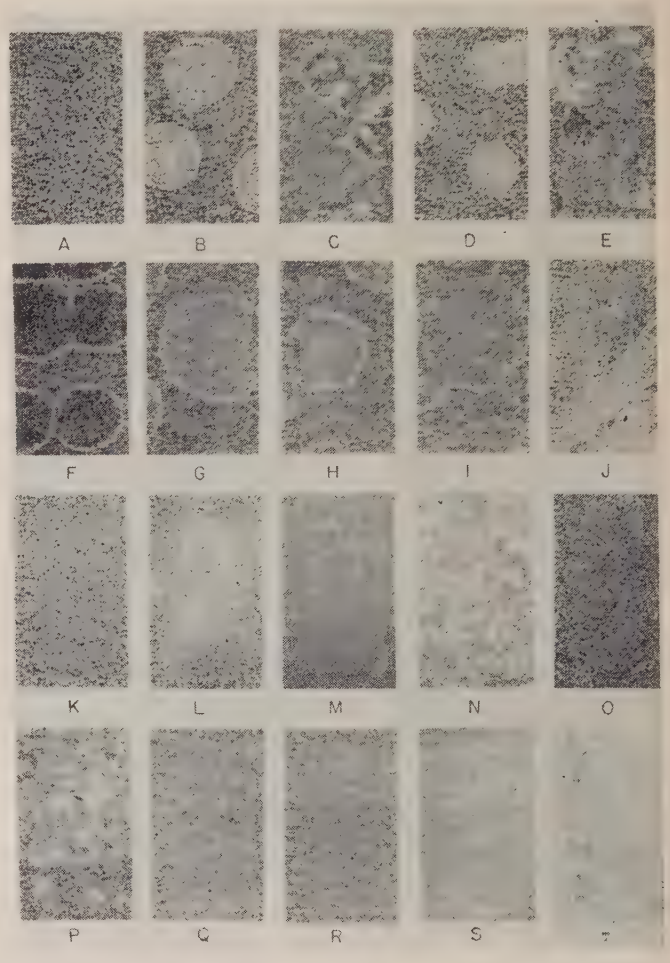



***Fig. 5. Surface wettability patterns. Vacuum—30 mm. Hg; backing—porous; speed—91 f.p.m.***

The degree of vacuum on the applicator was found to be extremely important in producing a picture of surface irregularities. Originally, the removal of excess ink from the paper surface was thought to be the sole action of the vacuum. Investigation revealed, however, that another phenomenon, which has been called the "impregnating effect," is very important. When a test run was made with maximum vacuum and a nonporous backing material, the ink was forced into the sheet and some ink penetrated completely through the sheet. With the same high vacuum but with a porous backing, the ink did not penetrate the sheet. Slight impregnation, however, did occur, so that the pattern was not the surface pattern of the paper (see Fig. 4). These observations revealed a new principle in the application of a vacuum to a porous surface, the basis of which is that a vacuum has an appreciable effect on the same surface to which it is applied and at an appreciable distance from the area of application. This principle shows that a fluid material (ink, coating color, size, wax, adhesive, latex, filler, etc.) can be forced into a porous surface by applying a vacuum to an adjacent area of the same surface.

The mechanism of the impregnating effect is simply that of continuously evacuating air from the interstices of the sheet while applying a fluid to the surface of the evacuated sheet.

Because of the existence of the impregnating effect, an absolutely true surface wettability picture of a porous sheet cannot be obtained with the present apparatus. Any amount of vacuum must cause some evacuation of the sheet and, therefore, some forced ink penetration, whereas operating with no vacuum deposits an ink film so heavy that an appreciable thickness of paper is saturated. In actual operation, therefore, the wettability of the paper is not the only factor. The impregnating effect must be considered and minimized by adjusting the speed and vacuum.




***Figs. 6 and 7. Effect of various surface treatments***

All samples were treated and tested on the felt side. Any liquid added to the sheet during the treatment was completely evaporated on a sheet drier before testing.

Key to Figs. 6 and 7.

(A) original paper, no treatment; (B) benzene dropped on sheet; (C) ethyl alcohol dropped on sheet; (D) ethyl ether dropped on sheet; (E) water dropped on sheet; (F) pine oil dropped on sheet; (G) kerosene dropped on sheet; (H) capryl alcohol dropped on sheet; (I) hexyl ether dropped on sheet; (J) sheet very lightly brushed with lubricating oil; (K) sheet exposed to oil vapor from a hot plate; (L) sheet blasted with air containing oil vapor from a vacuum pump; (M) dust from laboratory air allowed to settle on sheet for about two weeks, excess dust blown off before testing; (N) sheet held in saturated steam, then dried; (O) sheet soaked in water, blotted, and tested while wet; (P) sheet heated on drier, a wet blotter placed on the surface, and the combination dried; (Q) sheet dried 15 minutes on drier; (R) sheet dried 30 minutes on drier; (S) sheet heavily calendered; and (T) sheet dipped in water and then heavily calendered until dry with hot rolls.

*Final Operating Conditions*

It is apparent from the foregoing discussion that the paper properties—porosity, smoothness, wettability, and, probably, softness and flexibility—as well as such operating variables as blade clearances, ink, speed of ink application, backing material, and degree of vacuum, are so interrelated that there is no fixed set of conditions under which the surface properties of all types of papers will be most truly revealed. However, as nearly constant conditions as possible were selected to give the best over-all results. These conditions were:

| | |
|---|---|
| Ink | a 0.5% solution of Methylene blue ZX in distilled water |
| Speed | 91 feet per minute |
| Backing | plate glass or a duplex porous-nonporous assembly of rubber dental dam and blotter stock |
| Clearance of blades | front blade 0.0015 inch; rear blade 0.0030 inch |
| Vacuum | this variable was changed between 0 and 700 mm. Hg but most papers were tested at 30 mm. |

These conditions are, however, more or less arbitrary.

## RESULTS ON VARIOUS PAPERS AND THE EFFECTS OF VARIOUS SURFACE TREATMENTS

The surface patterns produced on both the felt and the wire side of about 35 commercial papers, including various bonds, coating raw stocks, hanging stocks, coated papers, printing papers, and boards, were examined and no two patterns were found identical. The pattern often varied from sheet to sheet within the same sample. Without exception, the wire side of all sheets gave the most uniform coloring. Generally, this coloring was as uniform as might be expected on a sheet of paper but, in a few instances, some mottling was noted.

Examples of five samples are shown in Fig. 5. These samples and also paper 11 of Fig. 4 were supposedly the same kind of coating raw stock. The differences, however, were so great that the variations could be detected by ordinary mill tests. This series was selected as an extreme example of what can happen during the manufacture of a given grade of paper. As a rule, the variations are considerably less but they can still be detected with the wettability tester.

The samples of this series were ranked visually according to their degree of mottling. This ranking was compared with such standard tests as density, Thwing formation, Gurley porosity, Bekk smoothness, and fluorescent size time and little correlation was found. The physical test most nearly in agreement with the wettability ranking was the size test. This test, however, is not particularly sensitive and, therefore, probably will not show differences experienced in an average run of paper. The size tests on the felt and wire sides of several papers were nearly the same but the surface patterns were markedly different. This point is substantiated by the experience of one observer (*7*) in the use of Rowland's tester to evaluate coating raw stocks. He found that the results of the tester did not correlate with any standard paper test but that they did give an indication of the behavior of the paper in the coating process.

As examples of a few of the factors which can influence the surface properties of paper, 20 samples are reproduced in Figs. 6 and 7. The same groundwood hanging stock was used in every case and the felt side was treated and tested. Specimen A illustrates the pattern of an untreated surface. Specimens B, C, and D illustrate the effects of spotting by three organic solvents.

If drops of water were to fall on the sheet during its passage through the driers, a pattern such as sample E might be obtained.

The action of some antifoaming agents is shown by samples F, G, H, and I. The areas of sample F, which were touched with pine oil, show great improvement in uniformity and thus indicate one reason why pine oil is such a satisfactory papermaking chemical. Perhaps the use of pine oil in the white water to provide a uniformly wettable paper surface would be at least as advantageous as its use as a foam killer.

Samples J, K, and L indicate the effect of oil and oil vapor. Drops of some lubricating oils on water do not spread to films but, with mild agitation, are broken into many small drops which seem to form a pattern on the water surface similar to the oiled areas of sample J. The odor of oil vapor from hot machinery is often noticed around paper machines. Samples K and L show the effect that such vapor may have on the paper surface. This effect would, of course, be enhanced if a quantity of vapor were drawn into the sheet by the suction boxes or other equipment which applies a vacuum to the sheet.

Sample M shows the pattern obtained after dust from the laboratory air had settled on the paper. A very uniform coloring was obtained. The reason for this marked improvement may be the effect produced by rough surfaces in decreasing the apparent contact angle of a liquid on a solid if the true angle is less than 90° (*8*).

If steam impinges on the paper, results such as shown by sample N might be obtained or, if a portion of the drier felt is excessively wet when it contacts the sheet, the effect may be similar to that of sample P.

Sample O illustrates the pattern obtained when the sheet is tested while wet. This probably explains why good results are often obtained when a wet sheet is coated.

The effects of excessive drying are seen to be small from samples Q and R. Excessive calendering, however, has a very great influence, as seen from samples S and T.

Numerous other surface treatments can be conceived and each will probably influence the paper surface wettability. For example, repeated trials (*7*) have demonstrated that a gentle brushing of the surface with a dry, soft-bristled brush will often change the surface so that it is wet very uniformly.

## DETERMINATION OF THE QUANTITY AND THE DEPTH OF PENETRATION OF INK IN THE TESTED SHEETS

A knowledge of both the quantity and the distribution of the ink in a tested sheet would be of value not only in understanding the action of the applicator but also in characterizing the paper when tested under given conditions.

Several methods of evaluating printed areas have been developed (*9, 10*), but none of the techniques seemed to supply the information desired in the present work; therefore, a new procedure was sought. The equations of Kubelka and Munk (*11*), which show the relation between the incident, reflected, and transmitted light from a thin homogeneous layer of absorbing and scattering material, contained the desired factors of absorption coefficient and thickness of the material.

Considerable work has been done with these equations (*12–23*) but, in all cases, the material being tested was considered as a single homogeneous layer, The work, therefore, was not applicable to the present problem in which the sheet consists of two layers—one of colored paper and one of original white paper.

One of the equations of the original derivation [(*11*), equation 3, or (*12*), equation I] was

$$2SX\sqrt{a^2-1} = \ln \frac{(R - a - \sqrt{a^2-1})\,(R' - a + \sqrt{a^2-1})}{(R' - a - \sqrt{a^2-1})\,(R - a + \sqrt{a^2-1})} \quad (1)$$

where

$X$ = thickness of homogeneous layer in mils
$S$ = scattering coefficient in reciprocal mils. This coefficient relates to the optical scattering in the sheet
$K$ = absorption coefficient in reciprocal mils. This coefficient relates to the optical absorption in the sheet
$R$ = absolute reflectance of surface on which light is incident
$R'$ = absolute reflectance of background behind the sheet
$a$ = $(S + K)/S$

This equation can be applied to any thickness of any homogeneous material which scatters and absorbs light.

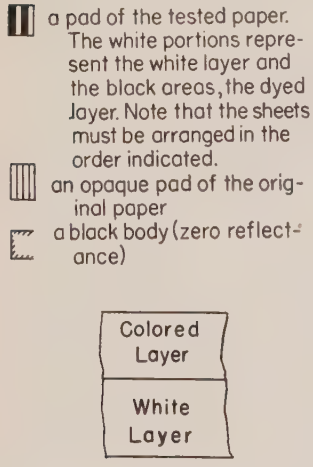

a pad of the tested paper. The white portions represent the white layer and the black areas, the dyed layer. Note that the sheets must be arranged in the order indicated.
an opaque pad of the original paper
a black body (zero reflectance)
Colored Layer
White Layer

*Fig. 8*

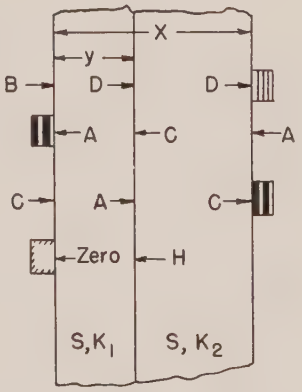



*Fig. 9*

Let us imagine that the cross section of a sheet colored with the wettability tester is as illustrated in Fig. 8. The above equation can be applied to the colored layer by having the light incident on the colored surface. The layer of white paper then serves as part of the background. The remainder of the background can be made up of a number of materials having known reflectances, such as a thick pad of the original paper, a black material having zero reflectance, or an opaque pad of the tested paper.

Or, the light may be made incident on the white side of the tested paper; then the equation can be applied to the white layer with the colored layer plus one of the materials above serving as the background.

With the mentioned general scheme of approach and the concept of the cross section of the sheet as shown in Fig. 9, several methods for determining the average quantity of dye in the sheet and the average depth of penetration of the dye were developed.

The following symbols are used in Fig. 9 and the following discussion.

$A$ = reflectance of the white side of a thick pad of the tested paper
$B$ = reflectance of the colored side of a tested sheet backed with an opaque pad of the original paper
$C$ = reflectance of the colored side of an opaque pad of the tested paper
$D$ = reflectance of an opaque pad of the original paper. The $R_\infty$ of previous workers
$H$ = reflectance at the interface of the two layers, of the colored layer backed with a material of zero reflectance (the $R_0$ of previous workers)
$y$ = thickness in mils of the colored layer. Average depth of penetration of the dye
$X$ = total thickness of the paper in mils
$S$ = scattering coefficient of the paper. This applies to both dyed and analyzed portions of the sheet, since it has been shown (*21*) that dyeing has little effect on the scattering coefficient
$K_1$ = absorption coefficient of the colored layer
$K_2$ = absorption coefficient of the original paper

By applying equation (1) to reflectance $B$ and to reflectance $C$, two relationships are obtained which can be equated and simplified to

$$\frac{K_1}{S} = \frac{A + B - C - D + ABC + ABD - ACD - BCD}{2(AB - CD)} - 1 \quad (2)$$

It is seen that reflectances $A$, $B$, and $C$ can be measured directly on the tested sheet. Likewise, $D$ and $X$ can be measured on the original paper and $S$ can be determined in the usual manner from one of the charts (*15, 18*) of the Kubelka-Munk equation. Therefore, from a thickness measurement and four easily determined reflectance values, the value of $K_1$, which is a function of the amount of dye per unit thickness of the colored layer for infinitesimally thin layers, can be calculated.

After solving equation (2) for $K_1/S$, the depth of penetration, $y$, of the dye can be obtained by directly solving the equation

$$y = \frac{\ln \dfrac{(B - a_1 - \sqrt{a_1^2-1})\,(D - a_1 + \sqrt{a_1^2-1})}{(D - a_1 - \sqrt{a_1^2-1})\,(B - a_1 + \sqrt{a_1^2-1})}}{2S\sqrt{a_1^2-1}} \quad (3)$$

which results from applying equation (1) to reflectance $B$.

The depth of penetration may also be determined by calculating the value of the reflectance of a thick pad of only colored layers—the $R_\infty$ of previous workers—from the following equation:

$$\frac{K}{S} = \frac{(R_\infty - 1)^2}{2R_\infty} \quad (4)$$

and the value of reflectance $H$—the $R_0$ value of previous workers—from the equation

$$H = \frac{D - B}{2a_1 D - BD - 1} \qquad (5)$$

and then using the Bureau of Standards charts (*15, 18*) to obtain the $Sy$ value of the colored layer. A simple division with the known value of $S$ gives the depth of penetration, $y$.

Equation 5 results from applying equation (1) to reflectance $H$ and reflectance $B$, combining the two resulting equations, and simplifying as before.

The above relationships are applied only to the present problem of ascertaining the amount and distribution in depth of dye in a sheet from the wettability tester. This is by no means the limit of application. Equation (2) is simply the relation between the ratio of two fundamental properties of a material and two reflectances of the material backed by two different materials of known reflectances. It may be applied, therefore, to any material under any conditions in which the reflectances of the backgrounds are known or can be measured.

## SURFACE ABSORPTION VALUE

The absorption coefficient, $K_1$, is a measure of the fractional amount of light absorbed per unit thickness of the colored layer for infinitesimally thin layers. Several workers (*13, 19, 21, 22*) have shown that the absorption effects of individual components in a sheet are additive. The product, $K_1y$, of the colored layer minus the product, $K_2y$, of an equivalent layer of uncolored paper is, therefore, proportional to the quantity of dye in the sheet.

$$(K_1 - K^2)y \approx \text{weight of dye} \qquad (6)$$

This value may be taken as a measure of the average wettability and absorption of the surface layers of a sheet. Inasmuch, however, as the coefficient, $K_2$, is usually very small as compared with $K_1$, it may be neglected and the value of $K_1y$ will satisfactorily represent the quantity of dye. This is particularly true with heavily colored sheets obtained with low vacuums on the wettability tester, which are the conditions used to best reveal surface wettability patterns. Hence, for the present work, $K_1y$ is suitable and has been designated as the "surface absorption" value.

In an examination of the impregnating effect at high vacuums, $K_1$ is generally less than 1.007 and $K_2$, which is of the order of 0.02, may not be neglected; therefore, $(K_1—K_2)y$ would probably be the most satisfactory measure of the degree of impregnation.

### *Results and Discussion of the Application of the Equations*

To obtain a series of papers with varying quantities and varying distributions of dye, a number of sheets of the same sample were run at several different degrees of vacuum. All other conditions were held constant. Reflectance values were measured with the General Electric recording spectrophotometer at the three different wavelengths of 540, 580, and 670 mmu.

The wavelength of light with which the reflectances are measured should, of course, markedly affect the value of $K_1$ but, if the relationships of the equations are true, the value of $y$ should be the same for all wavelengths. Actually, there appears to be a slight effect from 540 to 670 mmu. As the wavelength of light was increased, $y$ decreased about 0.07 mil or 2.3% of the thickness of the sheet. For the purposes of the present work, this change is negligible.

Two series of colored papers were made with different degrees of vacuum, one with a coating raw stock and one with a sulphite bond paper, on both the felt and wire sides of the papers. Calculation of the $y$ values showed that the depth of penetration reaches a minimum at between 10 and 30 mm. Hg vacuum. Apparently, at very low values, the vacuum is not sufficient to remove all excess ink from the surface. The remaining film is absorbed by the paper after the applicator passes on. As the vacuum is increased, the thickness of the film left on the paper reaches a minimum and then increases as the impregnating effect becomes more and more pronounced.

## RAPID ESTIMATION OF SURFACE ABSORPTION

Consideration of the derivation of equation (2) and of Fig. 9 indicates that either reflectance $C$ or reflectance $B$ might be a measure of the amount of dye in the tested sheet. The data which were secured are shown in Figs. 10 and 11. The points in the figures

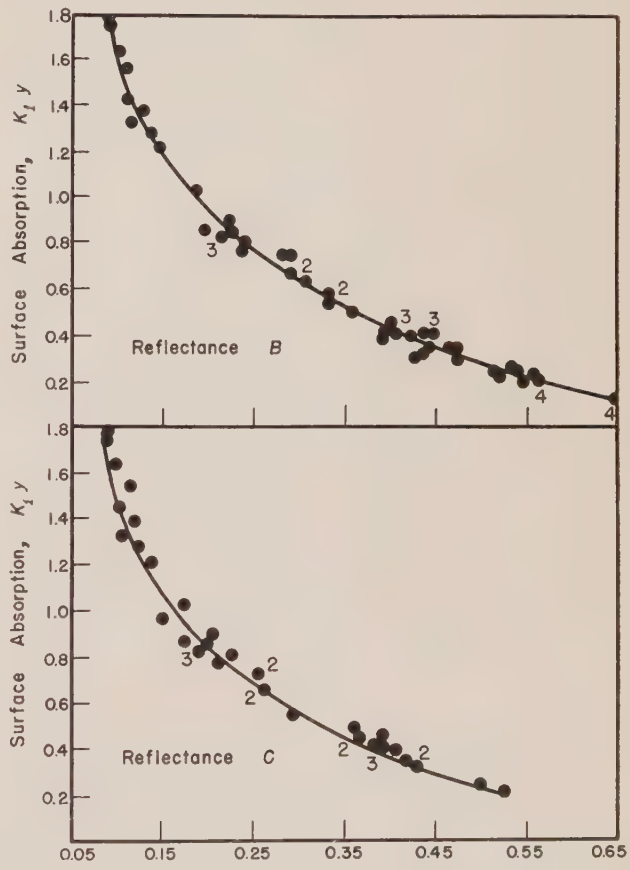



***Fig. 10.*** *(top) Relation of surface absorption to reflectance B*

***Fig. 11.*** *(bottom) Relation of surface absorption to reflectance C*

represent reflectances measured on both the felt and wire sides of a sulphite bond and a groundwood coating raw stock tested with vacuums from zero to 700 mm. Hg vacuum. Monochromatic light of wavelengths 540, 580, and 670 mmu, and tungsten filament light, were used. Even though the test conditions were so extremely varied, the data form a reasonably good curve in each case. It would seem likely that a fairly satisfactory measure of the surface absorption of a sheet could be obtained by determining either reflectance $B$ or $C$ of a sheet tested at a low vacuum and then reading the corresponding value of $K_1y$ from the appropriate curve. All test conditions of ink, speed, backing, vacuum, wavelength of light, etc. must be specified, of course, if values on different samples are to be compared. Inasmuch as the relations of Fig. 11 may have been fortuitous because of the types of papers used, construction of a specific curve for any given paper on which extensive work is to be done is recommended.

The tested sheet could also be examined visually for irregularities of surface wetting.

## SUMMARY

An apparatus which will reveal the surface wettability pattern of a sheet of paper was improved and constructed. The effects of operating variables were analyzed and a new principle of vacuum application, the impregnation effect, was discovered. Consideration of the mechanism of the action of the wettability tester shows that the true surface pattern of a paper cannot be obtained. With optimum operating conditions, however, a pattern sufficiently accurate for practical purposes can be obtained.

An equation of Kubelka and Munk (*11*) was extended to provide a means of measuring the thickness and the absorption coefficient of either layer of a duplex sheet in which both layers have the same scattering coefficient but different absorption coefficients and thicknesses. The derived equations were used to determine the average amount ($K_1y$) and the average depth of penetration ($y$) of ink in the colored sheet from the wettability tester.

Although the value of the absorption coefficient changed markedly with the wavelength of light used in measuring the reflectances, the value for the depth of penetration remained practically constant. Calculations based on reflectances measured with tungsten filament illumination are apparently satisfactory.

Two methods of examining the colored sheets were developed.

1. A simple visual examination was used to compare the mottling of one surface pattern with that of another, and thereby to compare the uniformity of spontaneous wettabilities of the surface. Usually, the patterns were quite different and could be ranked easily according to their degree of irregularity.

2. The fundamental quantity, $K_1y$, of the colored layer was calculated and used as a measure of the amount of a given dye absorbed by the paper under specified test conditions. This quantity was designated the "surface absorption" of the sheet.

Results of both types of examination did not correlate with standard paper tests.

A rapid method for measuring the approximate surface absorption of a sheet was illustrated. Only one reflectance of a tested sheet need be made; the surface absorption can be determined from a curve showing the relation between the reflectance and the quantity, $K_1y$.

Surface wettability and absorption are dependent upon chemical and physical properties of the paper surface and practically any type of treatment of the sheet (as a whole or of the surface only) will influence the surface absorption and alter the surface wettability pattern.

In view of certain practical experiences (*7*) and in consideration of the mechanisms involved, it is apparent that the surface absorption value, in conjunction with the visual examination of the surface pattern, will be an important factor in predicting the performance of a sheet in a coating, gumming, or aqueous printing operation more accurately than by any other known test.

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# A New Process for the Separation of Lignin and Cellulose

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**A new process is described for separating lignin and cellulose utilizing chlorine in the presence of boiling methanol. Methoxy lignin chloride derivatives of the lignin are readily recovered, substantially different from methoxylated and chlorinated lignins previously obtained. The methanol-chlorination process is limited by diffusion as are standard pulping processes. Although extractives in wood have no appreciable retarding effect on the reaction, moisture present must be limited to prevent conversion of the true chlorination reaction to an undesirable oxidative type of reaction. Semipilot plant operation in glass equipment indicates the possibility of accelerating the process by continuous countercurrent operation, yielding optimum delignification of a more resistant wood species, jack pine, in 1 hour at 60°C.**

Processes for separation of lignin and cellulose normally degrade both fractions. In research on various lignin reactions, it was found that chlorination of wood in methanol permitted the separation and recovery of a useful cellulose component; simultaneously producing a light-colored lignin product, inherently stable, yet reactive under controlled treatment.

Prior work has yielded chlorinated lignins from aqueous media (*2, 12, 20, 21, 22, 23, 30, 39, 40*). The oxidative type of reaction resulting in such media causes demethoxylation of the lignin product. Chlorination in organic solvents has been carried on at low temperatures (*10, 11, 17, 25, 26*), and although some workers believed that the presence of moisture was essential to the chlorination reaction, chlorination in methanol did yield partially methoxylated lignins.

Pulping of resinous species of wood raises difficulties which have been attacked with a variety of pretreatments (*3, 6, 7, 8, 9, 13, 28, 34, 35*), but practical applications have been limited. In the present work, effects of wood species, moisture content, particle size, and extractive content on the separation and nature of the lignin derivative were observed. Reaction rates and effects of temperature were also observed in the case of some wood species.

## PROCEDURE

Sawdust, shavings or small wood chips were agitated in boiling methanol in a three-necked round bottom flask, fitted with a chlorine sparger, condenser, and vent line to an absorber. Standard taper glass joints were used, and the agitator was sealed with acetylene tetrabromide. An electric hot plate supplied heat.

Methoxyl content of the lignin derivative was determined by Clark's modification of the Viebock and Schwappach method (*5*) with 1-hour reaction time. Chlorine content was determined on a separate sample by fusion with sodium peroxide in an electrically ignited Parr bomb; followed by precipitation, separation, drying, and weighing of chlorides as the silver salt. Alpha-cellulose was determined by the A.C.S. standard method (*31*).

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## METHOXY LIGNIN CHLORIDE STUDIES

Initial runs were made to determine the nature of the reaction and of the lignin products (designated MLC), using maple sawdust, methanol, and chlorine. Reaction temperature was the boiling point of methanol, 66°C. Following filtration from residual crude cellulose, MLC, which had been dissolved in the methanol, was precipitated (after concentration of the solvent) by admixture with water. Washing the residual crude cellulose (designated C-A) with hot methanol or dioxane yielded additional MLC after concentration and precipitation in water.

Data are given in Table I. Increase of reaction time from 2 to 3 hours (runs 5 and 6) increased MLC yield slightly, but decreased C-A yield considerably, indicating excessive decomposition of the latter. Run 7 gave comparable results showing that water was not essential for the chlorination reaction, as claimed by earlier investigators. Use of extractive free wood in run 12 indicated that extractives in wood had no adverse effect on the reaction, but probably exert some protective influence on cellulose subjected to acid degradation. Calcium carbonate in run 15 neutralized approximately half of the hydrogen chloride formed. The reduced yield of MLC and increased C-A yield showed that reaction was retarded; or acid cleavage is essential.

Methanol soluble MLC in Table I is substantially reproducible in the different runs; the dioxane soluble MLC, however, shows an appreciably lower chlorine content. In all later work methanol was used for this washing, and washings were combined with reaction liquor. Analyses of unpurified methanol soluble MLC from runs 5, 7, and 12 show good agreement.

| | *No. 5* | *No. 7* | *No. 12* |
|---|---|---|---|
| C | 43.4 | 42.5 | 40.8 |
| H | 4.6 | 4.0 | 3.8 |
| Cl | 28.0 | 28.1 | 29.5 |
| O (by difference) | 24.0 | 25.4 | 25.9 |
| $OCH_3$ | 22.4 | 20.8 | 19.8 |

MLC from run 12 should be the purest since extractive free wood was used. From this analysis, the formula of maple MLC might be

$C_{40}H_{28}O_{14}Cl_{12}(OCH_3)_9$—least molecular weight 1437.

Spruce MLC, assuming a molecular weight approximating that of maple MLC, has a chlorine content of 11 atoms, and a methoxyl content of nine groups. A spruce lignin derivative of Jansen and Bain (*17*) from a lower temperature reaction contained 13 chlorine atoms and seven methoxyl groups.

Maple MLC obtained from reaction in boiling methanol is completely soluble in methanol, acetone, ethyl

**Table I. Methoxy Lignin Chloride Studies—Preliminary Runs**

| Run | Wood, g. | Methanol, ml. | Time, hr. | Methoxy, yield, % | Lignin, $OCH_3$, % | Chloride, Cl, % | Crude cellulose, yield, % |
|---|---|---|---|---|---|---|---|
| 5 | 25.0 | 200 | 2 | 24.2 | m 22.4<br>d 21.6 | 28.0<br>.. | 73.3 |
| 6 | 25.0 | 200 | 3 | 26.4 | m 21.0<br>d 22.1 | 28.4<br>21.1 | 54.0 |
| 7[a] | 25.0 | 200 | 2 1/2 | 26.6 | m 20.8<br>d 21.4 | 28.1<br>25.0 | 62.3 |
| 12[b] | 50.0 | 500 | 2 1/2 | 24.7 | m 19.6 | 29.2 | 52.2 |
| 15[c] | 50.0 | 500 | 3 | 13.3 | ... | .. | 77.6 |

24-Mesh maple sawdust containing 6.0% moisture used in all runs except where noted.
m—Methanol soluble MLC.
d—MLC from dioxane washings.
[a] Anhydrous conditions. [b] Extractive free wood. [c] 14 g. calcium carbonate added to cook.

acetate, amyl acetate, and acetic acid; partially soluble in chloroform, carbon tetrachloride, ethanol, benzene, toluene, and carbon disulphide; and slightly soluble in ethyl ether, dilute sodium carbonate, dilute sodium hydroxide, dilute sodium sulphite, and ammonia, all at room temperature. Maple MLC reduces potassium permanganate, is acetylated by boiling with either glacial acetic acid or acetic anhydride, and is decomposed by boiling 10% sodium hydroxide. MLC is a cream-colored to light yellow amorphous, nonhygroscopic solid of variable melting point, susceptible to thermal decomposition.

### DIFFERENT WOOD SPECIES

Samples of various woods and bagasse were air-dried and chlorinated in boiling methanol. All runs were for 3 hours, except tamarack which was run for 5 hours. Data for products are given in Table II. Analytical data are incomplete for MLC from longleaf pine and Douglas-fir. Very similar MLC products were obtained from sugar maple, white oak, aspen, jack pine, eastern spruce, tamarack, and bagasse. Sugar pine yielded a highly methoxylated product, while redwood MLC was low in methoxyl. Yellow pine and white pine MLC's were low in chlorine, while redwood MLC had a high chlorine content.

Highest MLC yields were obtained from the most resinous species. The usual inhibitory effects of extractives on lignin separation are not evident; the extractives may have become part of the MLC product. Yields alone do not indicate the degree of delignification, because of variation in lignin and extractive contents of various wood species as shown by analyses (Table III). The total of lignin and extractives may be regarded as the total material potentially convertible to MLC. Degree of delignification is calculated two ways; first, on the basis of MLC recovered (after deduction of methoxyl and chlorine added; second, on the basis of C-A recovered. Both are subject to error; the former due to loss of MLC and extractives in dilute methanol solution, the latter because of loss of hydrolyzed cellulose fractions in water solution.

A wide difference between the two delignification values in Table II indicates excessive cellulose degradation, probably due to hydrolysis. The best checks were obtained for yellow pine, sugar pine, longleaf pine and redwood, some of the most resinous species. This would indicate that extractives protect the cellulose from hydrolytic reactions. On the basis of MLC yields, hardwoods and softwoods appear to react similarly, but there is considerable difference with regard to cellulose degradation.

### EFFECT OF EXTRACTIVES IN WOOD

It was noted previously that extractives probably do not interfere with the basic reaction but may have added to the yield of MLC without affecting its analysis. A more complete study was made on redwood, a highly resinous species. Extractives were removed from redwood shavings with a constant boiling mixture of methanol-benzene, and were chlorinated in this solution. MLC was recovered as usual.

Extractive free redwood was also chlorinated in methanol, as was a sample of air-dried, but otherwise untreated redwood. In order to establish a common time basis, a value for untreated redwood was inter-

**Table II. Methanol-Chlorination of Various Wood Species**

| Run | Wood | Yield of MLC, DWB, % | Per cent $OCH_3$ in MLC | Per cent Cl in MLC | Yield of C-A, DWB, % | Per cent delignification MLC basis | Per cent delignification C-A basis |
|---|---|---|---|---|---|---|---|
| | | Deciduous Woods (Hard) | | | | | |
| 48 | Sugar maple | 14.6 | 21.8 | 26.8 | 86.3 | 31 | 37 |
| 45 | White oak | 16.0 | 19.9 | 28.5 | 76.1 | 30 | 69 |
| P-8 | Aspen | 10.1 | 22.0 | 27.6 | 85.0 | 27 | 61 |
| | | Coniferous Woods (Soft) | | | | | |
| P-7 | Jack pine | 11.4 | 21.0 | 28.2 | 85.5 | 23 | 43 |
| 30 | Yellow pine | 24.1 | 20.4 | 23.4 | 85.0 | 41 | 37 |
| 32A | Sugar pine | 23.2 | 24.6 | 27.6 | 86.5 | 36 | 34 |
| 19 | White pine | 19.1 | 19.6 | 25.9 | 84.1 | 30 | 38 |
| 25 | Longleaf pine | 37.5 | .. | .. | 75.5 | 48 | 47 |
| 28 | Douglas-fir | 24.7 | .. | .. | 75.8 | 38 | 56 |
| 24 | Eastern spruce | 13.7 | 19.7 | 29.3 | 94.0 | 26 | 18 |
| 27 | Redwood | 46.2 | 16.2 | 32.9 | 72.6 | 50 | 49 |
| P-9[a] | Tamarack | 12.3 | 21.5 | 29.7 | 72.0 | 25 | 90 |
| | | Grass Fiber | | | | | |
| 77[b] | Bagasse | 16.5 | 21.4 | 29.4 | 68.4 | 42 | 100 |

[a] 5-hour run.
[b] Sugar cane waste.
DWB-drywood basis

**Table III. Wood Analyses**

| Species | Dry basis: Extractives, % | Dry basis: Lignin, % | Dry basis: Cellulose, % | Dry extractive-free basis: Lignin, % | Dry extractive-free basis: Cellulose, % |
|---|---|---|---|---|---|
| | | Deciduous Woods | | | |
| Sugar maple | 8.9 | 20.1 | 71.0 | 22.1 | 77.9 |
| White oak | 8.5 | 26.0 | 65.5 | 28.4 | 71.6 |
| Aspen | 4.2 | 20.5 | 75.3 | 21.3 | 78.7 |
| | | Coniferous Woods | | | |
| Jack pine | 4.9 | 29.0 | 66.1 | 30.5 | 69.5 |
| Yellow pine | 6.6 | 34.0 | 59.3 | 36.5 | 63.5 |
| Sugar pine | 9.9 | 30.4 | 59.7 | 33.7 | 66.3 |
| White pine | 12.5 | 29.6 | 57.9 | 33.9 | 66.1 |
| Longleaf pine | 24.3 | 27.8 | 47.9 | 36.6 | 63.4 |
| Douglas-fir | 13.1 | 30.0 | 56.9 | 34.5 | 65.5 |
| Eastern spruce | 6.6 | 27.6 | 65.8 | 29.6 | 70.4 |
| Redwood (shavings) | 24.7 | 31.2 | 44.1 | 41.4 | 58.6 |
| Redwood (Asplund fiber) | 9.6 | 29.3 | 61.1 | 32.4 | 67.6 |
| Tamarack | 6.3 | 25.7 | 68.0 | 27.5 | 72.5 |
| | | Grass Fiber | | | |
| Bagasse | 6.7 | 18.9 | 74.4 | 20.3 | 79.7 |

polated from the time curves given later. Data for yields and analyses of MLC and for yields of C-A are given in Table IV.

The high MLC yield and chlorine and methoxyl contents should be noted for the product of the extractives run. After correction for chlorine and methoxyl added (the latter estimated) it is seen that more than 50% of the redwood extractives are converted to a product closely resembling MLC in chemical and physical properties.

MLC obtained from extractive-free redwood is higher in methoxyl and lower in chlorine content than the extractives product. MLC from normal redwood has a chlorine content between the values obtained in the other two runs, but methoxyl is lower than either of the other values.

Normal redwood shows delignification values of 50.5 and 52.4% on basis of MLC and C-A, respectively. Extracted redwood shows 34.1 and 71.2% delignification on the same bases. This further indicates the protective influence of extractives on cellulose during the reaction.

### EFFECT OF MOISTURE IN WOOD

Water has been shown as unessential to the methanol-chlorination reaction, but small amounts present in the wood and solvent did not affect the nature of the MLC. A quantitative study of the effects of water was made on extractive free redwood shavings. Raw materials in these runs were made anhydrous, then 0 to 25% by volume of water was added to the methanol before chlorinating for 4 hours. Data are shown in Table V and Fig. 1.

Figure 1 shows the rapid decrease in MLC yield with increase in water content, while the apparent C-A yield increases indicating poorer delignification with increasing water. Methoxyl content of MLC decreases steadily with increasing water, while chlorine content first increases, then decreases. Assuming a molecular weight for redwood lignin of 840, and a methoxyl content of 15.8%, the number of original methoxyls is four.

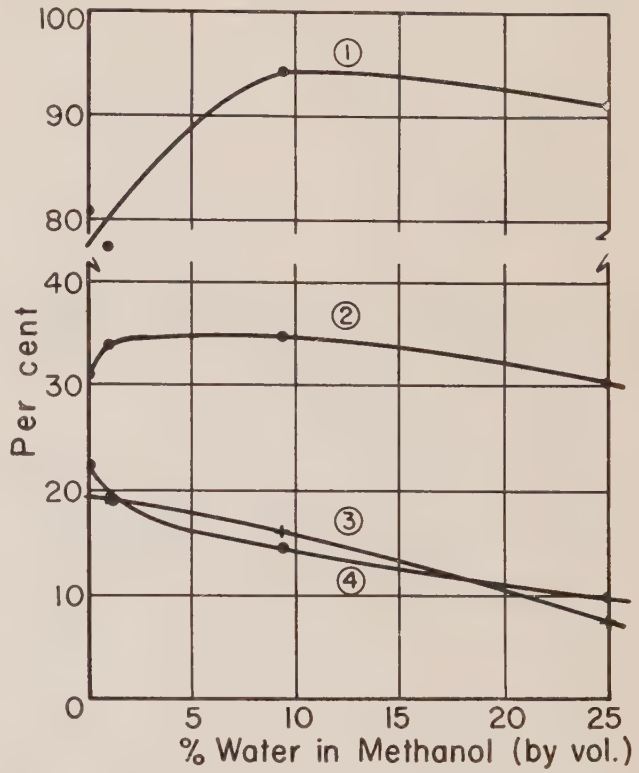


***Fig. 1. Effect of moisture on methanol-chlorination of redwood***

Curve 1 C-A yield, 2 chlorine content of MLC, 3 methoxy content of MLC, and 4 MLC yield.

In methanol chlorination, 0 to 1% water has no effect on methoxyl addition, which is five groups, for a total of nine; but chlorine addition increases slightly from 13 to 14 atoms per lignin molecule. Increase in water

**Table IV. Extractive Variable**

| Run | Material | Time, hr. | Yield of MLC, DWB, % | Per cent $OCH_3$ in MLC | Per cent Cl in MLC | Yield of C-A, DWB, % |
|---|---|---|---|---|---|---|
| [a] | Redwood | 4 | 49.1 | 16.1 | 35.5 | 70.6 |
| 52 | Extracted redwood | 4 | 19.2[b] | 19.5 | 34.0 | 77.8 |
| 58 | Extractives | 4 | 103.3[c] | 18.8 | 37.7 | .. |

[a] By interpolation of data from Fig. 4.
[b] On basis of dry extractive-free wood.
[c] On basis of extractives charged.

**Table V. Redwood Methanol-Chlorination—Moisture Variable**

| Run | Volume water in methanol, % | Yield of MLC, DWB, % | Per cent $OCH_3$ in MCL | Per cent Cl in MLC | Yield of C-A, DWB, % |
|---|---|---|---|---|---|
| 85 | 0.0 | 22.4 | 19.6 | 31.3 | 81.0 |
| 52 | 1.0 | 19.2 | 19.5 | 34.0 | 77.8 |
| 86 | 8.9 | 14.3 | 16.1 | 34.8 | 94.1 |
| 87 | 25.0 | 10.0 | 7.7 | 30.3 | 91.0 |

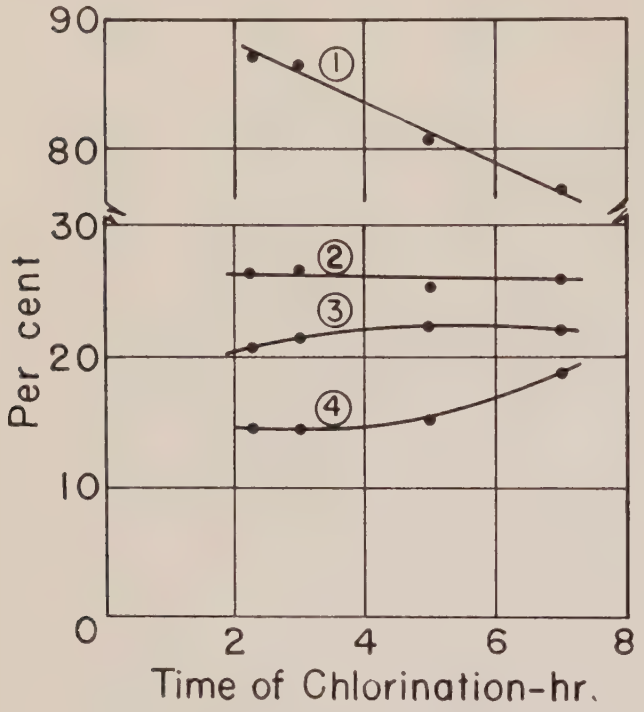


*Fig. 2. Effect of time on methanol-chlorination of sugar maple*

Curve 1 C-A yield, 2 chlorine content of MLC, 3 methoxy content of MLC, and 4 MLC yield.

above 1% decreases methoxyl addition, finally causing methoxyl removal; at 25% water in methanol, MLC contains only three methoxyl groups. Simultaneously, chlorine addition is reduced, amounting to only nine atoms per lignin molecule.

## REACTION RATE STUDIES

Sugar maple, white oak and redwood shavings, and jack pine Asplund fibers were used to study the rate of the basic reaction. Since methoxylation, chlorination,

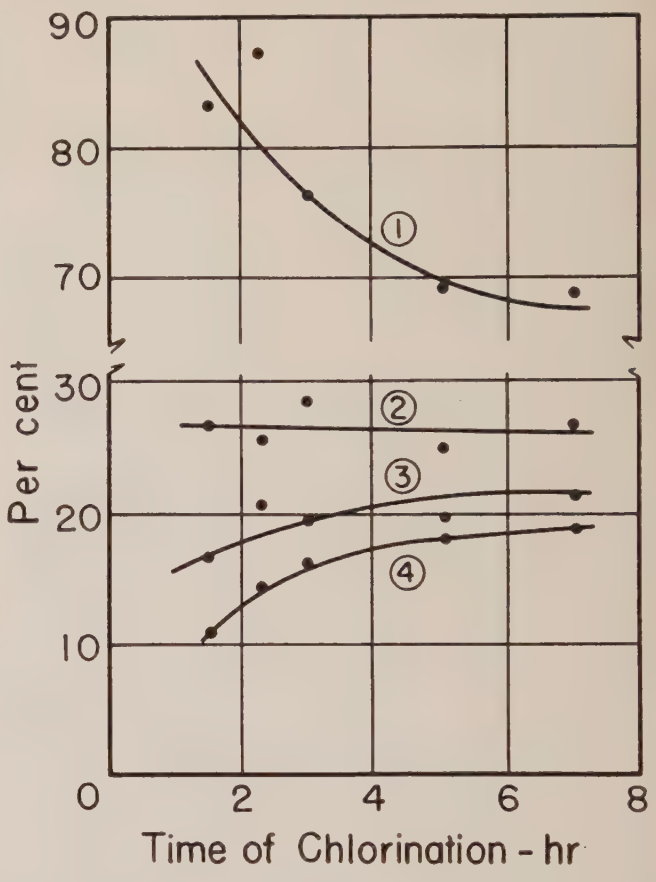


*Fig. 3. Effect of time on methanol-chlorination of white oak*

Curve 1 C-A yield, 2 chlorine content of MLC, 3 methoxy content of MLC, and 4 MLC yield.

**Table VI. Reaction Rate Variables**

| Run | Time, hr. | Yield of MLC, DWB, % | Per cent $OCH_3$ in MLC | Per cent Cl in MLC | Yield of C-A, DWB, % | Per cent delignification MLC basis | Per cent delignification C-A basis |
|---|---|---|---|---|---|---|---|
| | | | Sugar Maple Shavings | | | | |
| 44 | 2¼ | 14.6 | 21.0 | 26.2 | 87.2 | 35 | 44 |
| 48 | 3 | 14.6 | 21.8 | 26.8 | 86.3 | 35 | 47 |
| 43 | 5 | 15.5 | 22.5 | 25.5 | 80.4 | 37 | 67 |
| 47 | 7 | 18.7 | 22.2 | 26.0 | 77.1 | 45 | 79 |
| | | | White Oak Shavings | | | | |
| 33 | 1½ | 10.7 | 16.4 | 26.9 | 83.5 | 23 | 48 |
| 34 | 2¼ | 14.1 | 20.5 | 25.8 | 87.6 | 29 | 36 |
| 45 | 3 | 16.0 | 19.9 | 28.5 | 76.1 | 32 | 70 |
| 38 | 5 | 18.0 | 19.4 | 24.7 | 69.4 | 37 | 89 |
| 36 | 7 | 18.4 | 21.4 | 26.6 | 69.0 | 37 | 90 |
| | | | Redwood Shavings | | | | |
| 49 | 1 | 18.6 | 12.8 | 29.7 | 71.9 | 23 | 50 |
| 50 | 1½ | 19.9 | 12.5 | .. | 72.2 | 25 | 50 |
| 35 | 2¼ | 39.2 | 14.2 | 33.7 | 72.0 | 46 | 50 |
| 27 | 3 | 46.2 | 16.2 | 32.9 | 72.6 | 54 | 49 |
| 40 | 5 | 50.2 | 16.0 | 36.2 | 65.5 | 54 | 62 |
| 37 | 7 | 55.0 | 15.9 | 36.7 | 68.1 | 59 | 57 |
| | | | Jack Pine Asplund Fiber | | | | |
| 64 | 2¼ | 20.5 | 19.8 | 28.0 | 84.0 | 46 | 47 |
| 63 | 3 | 20.7 | 19.6 | 29.4 | 82.7 | 47 | 51 |
| 62 | 5 | 25.2 | 22.9 | 31.6 | 82.8 | 54 | 51 |
| 73 | 7 | 25.2 | 23.3 | 31.4 | 81.3 | 54 | 55 |

and solvolytic reactions occur simultaneously, the time rates could not be determined exactly. Furthermore, observation of yields and analyses of ligneous and cellulosic products are not reliable as measures of reaction rates because of losses of part of these materials in solution. Chlorine consumption is unreliable as a measure because of the large amounts absorbed by the solvent and wood, some of which is utilized in side reactions with the solvent.

Measurements were made of variation with time of yields of MLC and C-A, and analyses of the former; data obtained are shown in Table VI and Figs. 2, 3, 4, and 5 for the four wood species studied.

For sugar maple, C-A yields decrease more rapidly than MLC yields increase indicating degradation of cellulose. Chlorine content of MLC remains almost constant at 11 atoms, but methoxyl content increases with time to a total of 10 methoxyl groups (five in the original lignin—assumed molecular weight = 950, methoxyl content 20.3%).

White oak C-A and MLC yields, as well as chlorine and methoxyl content of the MLC, show the same relation as the sugar maple products; 11 chlorine atoms are added to the lignin molecule as well as four methoxyl groups (calculated on a lignin molecule similar to maple lignin).

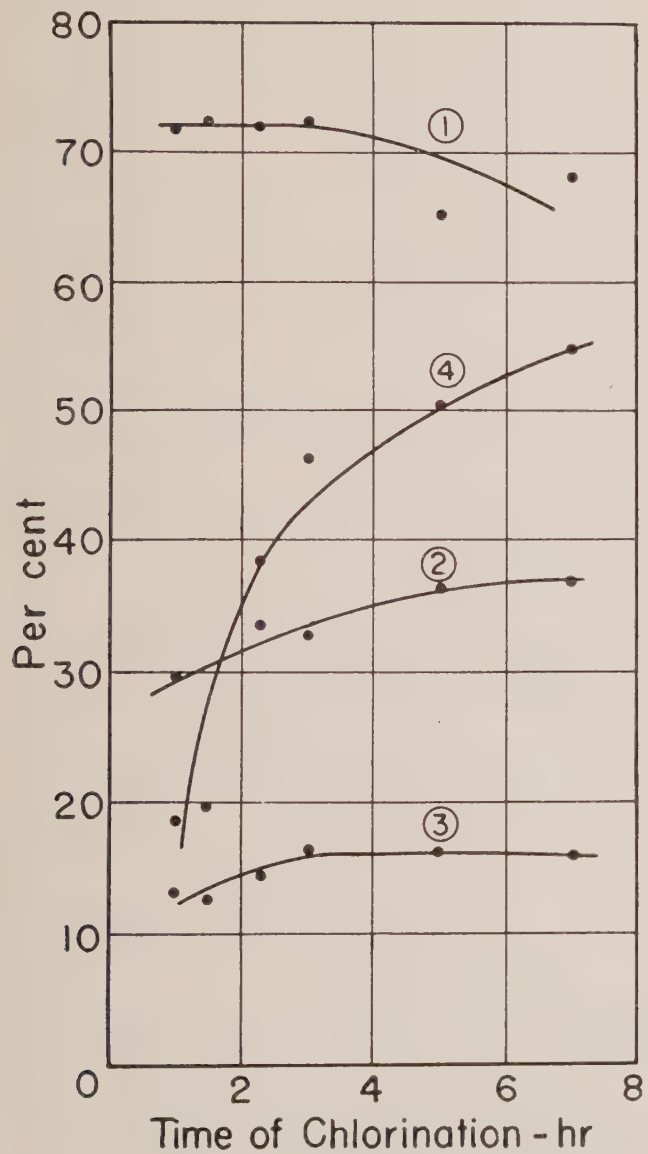


*Fig. 4. Effect of time on methanol-chlorination of redwood*

Curve 1 C-A yield, 2 chlorine content of MLC, 3 methoxy content of MLC, and 4 MLC yield.

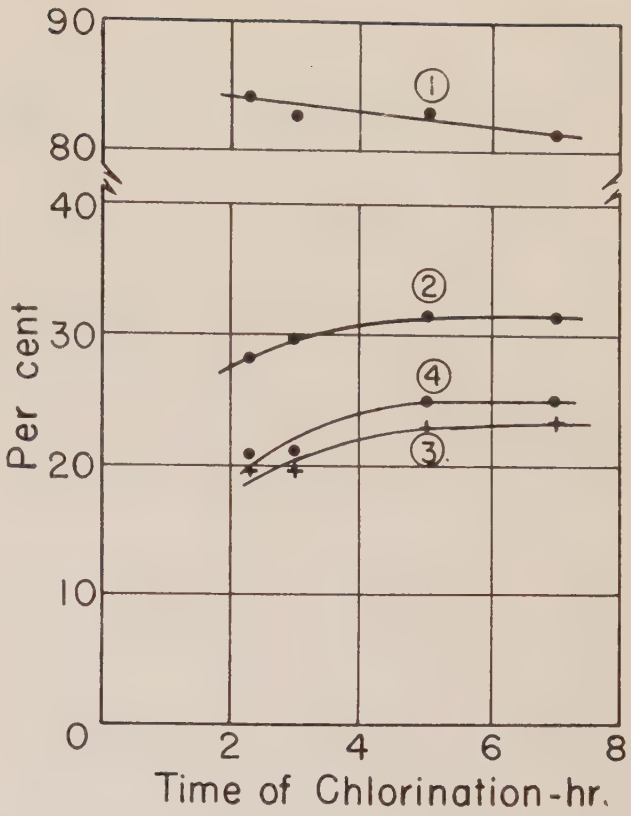


*Fig. 5. Effect of time on methanol-chlorination of jack pine*

Curve 1 C-A yield, 2 chlorine content of MLC, 3 methoxy content of MLC, and 4 MLC yield.

A different trend is noted for redwood, where MLC yields increase more rapidly than C-A yields decrease. This may be attributed to gradual conversion of soluble extractives to MLC, and the protective influence of these extractives on the cellulose fiber, as noted previously. Ultimate chlorine addition amounts to 16 atoms, while four methoxyl groups are added (based on an assumed redwood lignin molecular weight = 840, and a methoxyl content of 15.8%).

Jack pine shows more resistance to methanol chlorination than the other species, and excessive cellulose degradation results. Fourteen chlorine atoms are added to the lignin, as well as eight methoxyl groups (assuming original lignin similar to redwood lignin).

Analysis of reaction rate data indicates that the methanol-chlorination reaction is limited, like other pulping processes, by diffusion of reactants and products into and out of the wood fibers. An attempt to compare reaction rates indicated that the degree of delignification is an exponential function of time for the four species studied with jack pine showing a much slower rate of reaction than maple, oak, and redwood.

## SEMIPILOT PLANT EXPERIMENTS

In order to understand more fully the reaction and the possibilities of conducting it continuously, and to prepare larger quantities of MLC and cellulose for further experiments, a larger reaction unit was developed. This was constructed of standard Pyrex glass

pipe and fittings with a Pyrex centrifugal pump for circulating the solvent through a 5-foot section of 4-inch glass pipe. Various difficulties were overcome in the design and operation of this apparatus, and considerable information was assembled which would be useful in the development of larger scale equipment.

It was found that poor delignification was obtained when jack pine Asplund fibers were treated at 60°C. with an average time in the continuous system of $^1/_2$ hour. A 1-hour reaction time gave good results and $1^1/_2$ hours gave some degradation due to the hydrogen chloride present in the liquid. It was also found that temperatures above 50°C. are required for satisfactory delignification in a series of runs made with constant time and temperatures at different values between 47 and 61°. Various operating features indicated that higher temperatures and pressures would be desirable and would give shorter reaction times.

## CONCLUSIONS

The major process variables have been studied in laboratory and semipilot plant equipment to indicate the nature of the reaction and its applicability to various hardwoods and softwoods.

Although anhydrous conditions are unnecessary, limitation of water in the reaction is of critical importance. Water exceeding 3% of the reaction mixture will limit chlorination and methoxylation yielding an inferior MLC product. As methanol can be obtained and recovered at 99% strength, the wood used may contain 15 to 20% water or even more in a continuous countercurrent system where the solvent stream would have a drying effect.

Extractives in wood cause little difficulty. The extractives are evidently chlorinated and methoxylated in the same manner as lignin, and yield similar products. This indicates that some of the extractives are related to lignin in composition, or are intermediates in the plant synthesis of lignin. There is also evidence that the extractives protect the cellulose from excessive acid degradation during initial stages of the process. Wood species such as redwood and longleaf pine, which are resistant to standard pulping processes because of their high extractive content, are delignified readily by this process.

Hardwoods and softwoods, as well as bagasse, may be delignified. A few species are more resistant and give excessive cellulose degradation. Interesting color changes were noted for various wood species during the reaction; ranging through red, purple, and even black before the normal yellowish color of the crude cellulose is attained.

The reaction rate is similar for redwood, sugar maple, and white oak, but for jack pine it is considerably slower.

Semipilot plant operation showed that continuous counter-current operation resulted in major delignification in 1 hour at temperatures of 50 to 60°C. It was indicated that operation at elevated pressures and temperatures would permit further reduction in reaction time.

Semipilot plant work served only to indicate the feasibility of continuous operation of the process. Further development will require the use of equipment permitting operation at moderate pressures.

Measurements of chlorine and methanol consumption in the laboratory and semipilot plant runs were not subject to satisfactory control. Evidences of side reactions were noted; the formation of an explosive flame where chlorine entered the methanol in the presence of light of high intensity (such as direct sunlight), and the collection of a volatile fraction having the physical properties of methyl hypochlorite. The flame problem was overcome by proper light shielding. Analytical data indicate that methyl hypochlorite is an active intermediate, and this material must be retained in the system.

Economic analysis of this process is dependent upon further pilot plant investigation, and more complete evaluation of the methoxy lignin chloride and cellulose.

The process described above is covered by U. S. pat. 2,491,499.

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# Electrification of a Pulp and Paper Mill

J. C. LYNCH

**The electrification of a pulp and paper mill which started operations in 1899 is described. With the exception of a few initial years of operation, electric power has been utilized for auxiliary drives from the earliest days of the company's pulp and papermaking activities, and as far back as 1917 the program of electrification of main drives was started. Today the plant operates a 21,000-kw. central power station to serve a pulp and paper mill with an average daily output of 300 tons, completely electrified and driven by approximately 1500 motors totaling 28,000 hp. The history of this generating station and the growth of the papermaking facilities and paper production which made it necessary are traced.**

THE PLANT began operations in 1899 with a two-machine mill, including complete wood room and pulp cooking facilities using the sulphite process.

In the interval between 1899 and 1903 no electric power was generated or purchased and all power requirements were developed by steam engines.

A vertical steam engine-driven pump was used for the main water supply. A 1500-hp. Allis-Corliss cross compound condensing engine was used to drive the beater room and all equipment in the pulp mill including the wood room. The paper machines and auxiliaries were driven by steam engines, as were heating fans and other miscellaneous equipment.

As the mill expanded, more engines were added for paper machine and pulp mill drives. In 1907, the Allis-Corliss engine was relieved of the pulp mill load and used exclusively for beater drives, plus a belt connected direct-current generator. Simultaneously, the first Hannover-Lentz poppet valve engine installed in this country was put in service to drive the wood room and pulp mill. It was rated 750-hp. tandem condensing. Supercalenders were installed in 1911 and driven by a Lentz engine. In this same year, the first turbine-driven mill water pumps were installed. In 1917 two lineshaft-driven ammonia refrigeration compressors were installed in the acid plant.

With all initial power requirements being served by steam engines, it was natural that the original boiler plant and engine room should be located in the center of the plant between the pulp mill and the paper mill. The initial boiler plant installation consisted of five 150-p.s.i. units with a total capacity of 72,000 lb. per hr. In the interval 1899–1913 this plant was expanded to a total of 11 units with a combined capacity of 158,000 lb. per hr. After the present power house was built in 1917, in a new location, these 150-p.s.i. boilers were gradually retired from service. The last units were removed in 1946.

The first electric generators were 250-volt d.c. units. The first, a 150-kw. lineshaft-driven machine, was installed in 1903 and used on lighting service only. As auxiliary drives were motorized commencing in 1905, additional steam engine-driven d.c. generators were added until, by 1911, there were four units with a total capacity of 500 kw. (two 150-kw., two 100-kw.). These generators remained in service in the original main engine room until 1930 when a 2300-volt, 200-kw. motor-generator set replaced one 150-kw. and one 100-kw. unit; a second 200-kw. motor-generator set replaced the remaining two engine-driven generators in 1942. These two 200-kw. MG sets are now in service located in the central station, and supply all of the rather limited direct-current constant potenial requirements.

The first application of electric motors was on paper machine rewinders in 1905. These motors were double-ended units with two windings on the armature and a commutator at each end of the shaft. They were designed for series-parallel operation of the two armature windings and, when used with a drum controller, provided a speed range of 10 to 1 on the rewinder. Several of these motor frames are in service today although the armatures have since been converted to conventional single winding and single commutator units.

The direct-current constant potential system has always been limited to auxiliary drives and never expanded beyond the size it attained in the first 8 or 10 years of its service. From figures previously quoted it can be seen that the present day direct-current system is smaller in capacity than the system of 35 years ago. Today individual direct-current drives are limited to rewinders, cutters, fourdrinier shake drives, savealls, washers, and a few miscellaneous applications with the trend pointing to still further load reductions.

The present alternating-current generation and distribution system was started in 1914 with the installation of a 750-kva., 480-volt, 60-cycle condensing turbogenerator in the original main engine room. This alternator and a 600-kva. back-pressure turbogenerator installed alongside in 1934 were the only 480-volt alternators ever used. Later alternator installations were 2.4-kv. and 13.8-kv. units, and the 480-volt system was served largely through transformers and unit substations. Today the 750-kva. machine is idle and the 600-kva. unit has recently been retired from service and sold.

By the year 1917 the mill had grown to a five-machine mill following the addition of three paper machines in 1903, 1911, and 1912. All paper machines and main drives were steam-engine driven and the use of electric power was still confined to lighting and auxiliary drives. In view of the growth of the mill, it was decided to expand both the steam and electrical generation facilities. The new facilities were installed in a new building on the lake shore and formed the nucleus of the present central station.

Four new 250-p.s.i. boilers with chain-grate stokers

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were installed; total capacity of these units is 90,000 lb. per hr. These boilers are in service today although their use is somewhat restricted due to the addition of later facilities. The initial electrical generator in this new station was a 2000-kw, 0.75-p.f., 2.4-kv., 60-cycle condensing turbogenerator, the first 2.4-kv. unit in the plant electrical system.

In order to tie the new 2.4-kv. generator with the existing 480-volt system a 1500-kva. transformer bank was installed at the main engine room where the 480-volt alternator was located. Although the 480-volt alternators are no longer utilized, this transformer installation, later increased to 2000 kva., is still in service and the main engine room has become a 480-volt substation serving the paper mill area.

In 1920 the demand on the power system had increased to the point where it was felt necessary to purchase power from the local utility. This arrangement lasted for three years during which time the plant purchased about 12% of its total requirements. In 1923 two additional 2000-kw. generators were installed, duplicates of the original 2000-kw. installation. Since that time the plant has generated all of its power.

The availability of a large block of 2.4-kv. power made possible the electrification of some of the larger drives.

In 1920 several jordans were equipped with 300-hp. direct connected induction motors. The drives proved to be overmotored and affected the power factor adversely; in time, they were replaced with 200-hp. direct connected synchronous motors.

Mill water pumps were first motorized in 1917; by 1923 the electrification of these pumps had been completed, with 150-hp. induction motors replacing the turbines. In 1926, the engine driving the refrigeration plant compressors was replaced with two 75-hp. slip-ring motors; expansion of the plant included a third compressor driven by a 200-hp. direct connected synchronous motor. The supercalender engine was replaced in 1929 with a 125-hp. synchronous motor.

The 750-hp. Hannover-Lentz engine driving the pulp mill and wood room lineshafts was retired in 1930. The individual lineshafts were motorized; today the wood room and bleach plant lineshaft are still in operation but the majority of pumps and all of the flat screens in the pulp mill area have been individually motorized.

The first paper machine drives to be electrified were Nos. 2 and 5, in 1928 and 1931, respectively. Both machines were equipped with sectional drives and, in both cases, the direct-current generator was steam engine driven. In the case of No. 2, a decision to raise the top machine speed from 500 to 800 f.p.m. would have necessitated a complete new mechanical drive. A sectional electric drive was selected in preference to the mechanical drive in spite of the somewhat higher cost of the former, primarily for the sake of obtaining experience with this type of drive. In the case of No. 5, the machine was rebuilt to include additional drying capacity. Because of the physical location of the machine and the drive space limitations, a sectional electric drive was installed. Although, in both cases, the main generator was steam engine driven, a complete synchronous motor-generator set was installed, looking forward to the day when the drive would be completely electrified.

As production and electric power demands increased a major extension to the central station was made in 1936. Two boilers were added, each rated 675 p.s.i. and 80,000 lb. per hr. The station's fourth turbogenerator was installed, a 4000-kw., 0.70-p.f., 2.4-kv., 60-cycle machine. The turbine is a backpressure unit with steam supply at 675 p.s.i., exhausting at 150 p.s.i. The addition of this alternator raised the short circuit capacity of the electrical system beyond the interrupting capacity of the station switchgear; consequently a new 2400-volt switchroom, with larger circuit breakers, was included in the expansion. The boiler auxiliary drives were motor driven. At this time a second 2000-kva. transformer bank, 2400/480 volts, was installed to serve the pulp mill area, and the original 2000-kva., 480-volt substation was confined to paper mill area service.

Beater room electrification occurred in 1938, precipitated by failure of the crankshaft on the steam engine driving the beater room lineshaft. The lineshaft was split into several sections and each section was motorized. In a short time this arrangement was replaced by individual 75-hp., 2.4-kv. drives.

Number 6 paper machine was installed in 1940 with a single motor lineshaft drive. This was the first paper machine to be installed with the drive initially electrified.

Following the beater room electrification the only remaining steam engine drives were on the paper machines. In 1941 these engines were retired and the machines completely electrified. Numbers 2 and 5 had been previously equipped with electric sectional drives; on Nos. 1, 3, and 4 paper machines the lineshafts were retained and the machines equipped with a single motor drive.

The fifth turbogenerator was installed in the central station coincident with the electrification of the paper machines. It is a 5000-kw., 0.80-p.f., 2.4-kv., 60-cycle unit; steam pressure at the turbine throttle is 150 p.s.i.; the unit exhausts at 35 p.s.i. Installation of this unit was required because of increased electrical load and low pressure steam demand.

While no expansion of power generation or papermaking facilities occurred during the war years, plans had been laid for a postwar program. In 1945 this program commenced and included new steam and electric power generation facilities, a hydraulic log barker, a new and larger paper machine, pulp preparation facilities, and a processing machine for air-dried papers.

Two new boilers were installed, each rated at 675 p.s.i., 150,000 lb. per hr.; simultaneously, the last of the 150-p.s.i. boilers in the original boiler plant were retired from service and removed to make way for the new paper machine. The new turbo-alternator was a radical departure from previous installations: the alternator is a 7500-kw., 0.80-p.f., 13.8-kv., 60-cycle unit; the turbine is a 675-p.s.i. condensing unit with two stages of extraction, at 150 p.s.i. and 35 p.s.i.

The 13.8-kv. generator was tied to the existing 2.4-kv. system through a 6000-kva. tie transformer; in addition, two 13.8-kv. feeders were installed to serve unit substations in the new paper machine area.

The increase in station generating capacity again raised the short circuit capacity of the system beyond the interrupting capacity of the 2.4-kv. switchgear. The 2.4-kv. bus was then split into several sections and tied to a synchronizing bus through reactors. Individual reactors were installed in the 2.4-kv. generator and feeder circuits and the short circuit capacity was reduced to meet the interrupting capacity of the switchgear.

The hydraulic barker pumps and the pulp processing beaters were all equipped with 2.4-kv. drives; the paper processing unit required only 480 volt service.

The new No. 7 paper machine is the largest to be installed in the plant. It is driven by a single generator electric sectional drive with separate starting and running generators. The main drives and all auxiliary drives are powered from unit substations fed from the 13.8-kv. system.

It is expected that existing power facilities will accommodate normal plant growth for a number of years to come. It is not likely that a physical expansion of the central station will be required in the near future; any expansion of steam or electric facilities would be likely to follow the line of replacement of existing 250-p.s.i. boilers with 675-p.s.i. units and the replacement of the 2000-kw., 2.4-kv. alternators with 13.8-kv. machines, including expansion of the 13.8-kv. distribution system.

## ELECTRICAL DISTRIBUTION

In a normal year of three hundred days operation, the plant utilizes approximately 100,000,000 kw.-hr. of electrical energy. This compares with a total of 9,000,000 kw.-hr. used in 1919, the first full year of operation with the 2.4-kv. system. Of the total power generated, the following percentages are utilized for the services indicated:

| | | |
|---|---|---|
| 1. | Power plant auxiliaries | —12% |
| 2. | Mill water pumps | —11% |
| 3. | Pulp mill | —22% |
| 4. | Paper mill | —47% |
| 5. | Finishing | — 4% |
| 6. | Miscellaneous | — 4% |
| | Total | —100% |

Although almost 50% of the power is used in the papermill, the greater part of this power is expended in stock preparation and stock and water handling rather than on the paper machine drives themselves. It is significant that the ratio of pulp and paper production to power consumption has consistently averaged a figure of 2 lb. per kw.-hr. for the past 30 years (total kilowatt-hours includes electrical generation plus equivalent steam-developed power).

The original alternating-current system voltage was 480 volts. The size of the first alternator in the present central station, 2670 kva., dictated the change to a 2400-volt system; the size of motor required for main drives, as large as 300 hp., indicated the use of 2300-volt motors. A line was arbitrarily drawn at the 50-hp. level; all motors below this rating were to be 440-volt motors, and all motors 50 hp. and above were to be 2300-volt units. This voltage distinction was maintained until the advent of the unit substation when it became more economical to use 440-volt motors in ratings up to and including 150 hp.

The increase in generation voltage from 2.4 to 13.8 kv. was again dictated by the size of the last central station alternator, 9375-kva., and switchgear requirements for a machine of this size.

Varnished cambric, lead covered cables have always been used for the high-voltage feeders; the majority of the installations are in underground ducts, with a few in exposed conduit. The 2.4-kv. cables are for the most part single conductor, running as large as 750 MCM.; the 13.8-kv. cables are three conductor, 350 MCM. The system has been remarkably free of cable trouble, and there has never been an instance of cable failure.

The 2.4-kv. system is ungrounded; the 13.8-kv. system is grounded through a neutral resistor at the alternator. Relay protection includes differential relays on the 13.8-kv. and the larger 2.4-kv. alternators while no relay protection is used on the three 2000-kw. machines. Feeder circuits are each equipped with two induction type overload relays with instantaneous trip attachments.

Feeder outages have averaged slightly in excess of one per month for the past five years. None of the feeder circuits are equipped with automatic reclosing equipment, although instructions to the station operators call for two immediate reclosures in the event a feeder breaker trips. A check of feeder outages in this interval indicates that at least 75% of them could have been avoided had the circuit breakers been equipped with automatic reclosing devices, properly coordinated with time delay undervoltage protection devices on individual motor drives.

The first unit substations were installed in 1947 to serve the new paper machine and pulp processing facilities. Two 1500-kva., 2.4-kv. and five 750-kva., 480-volt units were installed on the 13.8-kv. distribution system. Two 13.8-kv. feeders are brought to each substation; a 13.8-kv. interrupter and selector switch is used to switch the transformer between feeders or de-energize it. Each unit is supplied with complete metering facilities, a secondary circuit breaker, and the requisite number of individual motor or feeder circuit breakers. A careful analysis of present and anticipated load requirements is of the utmost importance in obtaining maximum economy in unit substation installations.

## CONCLUSION

The largest single electrical problem in a pulp mill and paper mill is the problem of corrosion and contamination caused by water, acid, and filler. The problem is primarily a maintenance problem but much can be done in the design of electrical installations and the application of equipment to reduce maintenance and replacement costs. The proper motor enclosure, the location of conduit lines, and the location and ventilation of motor control equipment are all major factors to be considered in installation design.

The growth of an electrification program must be accompanied by a growth and expansion of maintenance facilities and personnel. Since the electrification of paper machine drives has occurred within the

memory of most of the operators, they are extremely critical of the performance of the electric drive. In order to obtain maximum results from the electrical equipment it is essential that a program of preventive maintenance be effected to insure continuity of service and performance.

The initial success of an electrification program can be insured by good installation design and proper application of equipment; the continued success of the program can be insured through a well-planned system of preventive maintenance.

### DISCUSSION

R. F. Sorenson (The Union Bag & Paper Corp., Savannah, Ga): Mr. Lynch's report was of particular interest to me. Those of us who have come into the paper industry in the past few years have seen a completely electrified mill and have not had a picture of the development that has taken place in the last fifty years. We have taken it all for granted without having any real conception of the work that had to be done by the older mills in bringing them to full electrification. Behind all of the achievements of Hammermill are a great many solved problems. Problems that, had they remained unsolved, would have prevented or greatly hindered the advancement of the program. These problems were solved one by one, and today much of the electrical system is taken for granted and known to be reliable.

We are in a position, however, to witness another phase of electrification that is growing and developing now. The demand for better speed regulation of our sectional paper machine drives and the demand for closer control in other operations have brought about the application of electronics.

The purpose of this discussion is to briefly outline the work at Union Bag in this phase of electrification. In 1947, we installed our No. 5 paper machine. This machine has complete electronic amplidyne control. This past Labor Day we converted No. 1 paper machine, and this Christmas we plan to convert our No. 2 paper machine to electronic-amplidyne control. The main purpose in this conversion is to make possible higher speed operation of these machines. As it was believed that electronic control would give much better speed regulation than the older selsyn control. The rated top speed of No. 1 machine was increased from 1200 to 1500 f.p.m. by changing the gear ratios in the speed reducers. The same change in gear ratios will be made on No. 2 machine. We are now in position to compare the operation of No. 1 machine with and without electronics, and the results have been encouraging. Normal speeds for the various weights of kraft paper run on this machine have been increased. We have run 40-pound kraft for considerable periods at 1500 f.p.m. Since the installation is comparatively new, we hope to better our present performance. The figure generally accepted for the speed variations in the old selsyn control was 0.1%. From measurements we have taken on the various sections of No. 1 machine, we now find our speed variations to be within 0.05%. Our speed variations have been cut in half, and the time of response of the control to sudden load changes has been decreased many times.

This conversion to electronics has not been without its difficulties. Our No. 5 machine, I believe was one of the first all-electronically controlled machines put out after the war. We had our share of control problems that had to be solved and after that we had still another problem. Mr. Lynch pointed out that in order for electrification to be a success, maintenance facilities and personnel must also grow. Soon after No. 5 went into production, we found that when electronic control got into trouble, the lost time was usually very high. Those men who were experts on the selsyn control were lost in electronics. We had a class for the operating electricians under way at the time to teach basic electronic theory and then how to apply it to the particular circuits in the paper machine control. These classes were successful enough but there was still lacking that knowledge one gets from being in trouble and then solving the problem. Some time was lost by assuming that the particular difficulty was electronic trouble and by going through an amplifier looking for it when it was not there. In general, it was a new type of control and the men were afraid of it. We actually had a comparatively small amount of trouble in the electronic circuits.

When No. 1 machine was converted, there was no high period of lost time due to electrical trouble. The electrical department had become used to electronics. They knew how to recognize the troubles and how to get out of them. We have progressed and are now accepting and taking for granted the equipment that two years ago was considered by many as experimental in this field. It must have been this way during many periods in those fifty years that Mr. Lynch has described. Those things that were acceptable were retained, and those things that were not practical are mainly forgotten.

In order for electronics to take its place in industry, its application must be made intelligently. If magnetic control can do the job just as well as electronic control, it is not easy to justify electronics. At present, its applications must be made only where very accurate control of speed or timing will bring about a definite improvement in quantity or quality of production. Electronic control, by the very nature of its ability to be very fast and accurate, is most susceptible to trouble. More thought must be given to its installation and care. If it is a correct application, all will be well worth while. There will always be borderline cases where it will be difficult to determine whether to apply electronics but it now seems certain that electronics has established itself in the control of sectional paper machine drives. Application of electronics throughout the entire paper mill and the bag factory seem to indicate that it has established itself in the industry and is now a part of any electrification program.

I have added these few remarks after Mr. Lynch's very complete report to indicate the experience we have had at Union Bag in one important application of electronics, and how a correct application of equipment and a capable group of maintenance men has led to the acceptance and extension of a comparatively new type of control.

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# Conversion of Mannogalactan Mucilages in Aqueous Borax Solutions for Use as Tub-sizing and Coating Adhesives

JOHN W. SWANSON

**The effect of borax upon guar and locust bean gum for their conversion into suitable papermaking materials was investigated. Several of the converted products were evaluated as tub-sizing and clay-coating adhesives in laboratory and mill scale trial with encouraging results.**

MANNOGALACTAN mucilages, such as guar and locust bean gum, possess outstanding adhesive properties for papermaking purposes when added to the stock prior to the formation of the sheet. It seemed reasonable to assume that such materials might also prove to be superior adhesives for tub-sizing and coating purposes. However, the natural products possess a very high viscosity which makes it necessary to convert or partially degrade them to a suitable viscosity before they can be used. Several satisfactory methods of doing this have been investigated but this report will be concerned only with the sodium hypochlorite oxidation of these mucilages.

During the early stages of the work, the matter of a suitable medium in which to carry out the conversion reaction became somewhat of a problem. Mannogalactans hydrate and dissolve readily in water, thereby making it difficult and expensive to isolate the converted products. Alcohols and other organic materials in which the products are not appreciably soluble are expensive and often unsuitable because of reaction with the converting agent. Attempts were made to use tertiary alcohols and carbon tetrachloride but these did not provide uniform reaction media and gave products of very poor color. Later it was found that an aqueous borax solution provided a suitable conversion medium. This procedure is an application of the property of mannogalactans to form a water-insoluble gel in the presence of borax. In this case, the small particles of mucilage do not dissolve because each particle is presumably surrounded with a layer of the borax gel, thereby preventing the penetration of a sufficient amount of water to disperse the mucilage. Following the conversion reaction, the mucilage may be filtered off, washed with dilute borax solution, and dried. This gives a fine white product having a much lower viscosity than the original product.

## EFFECT OF CONCENTRATION OF BORAX

One of the first factors investigated was the permissible range of borax concentration which could be employed from the standpoints of mucilage hydration and solubility. This was done by suspending 5 grams (4.22 grams oven-dry ash-free product) of locust bean gum in 100 ml. of the desired concentration of borax solution. The degree of swelling in milliliters was observed after standing 24 hours. The gums were then filtered off and yields determined. The data are presented in Table I and are plotted in Fig. 1.

The ash content increased steadily with borax concentration, whereas the degree of hydration or swelling was greatest in the lower concentrations of borax and seemed to approach an almost constant value at borax concentrations of 2.0 to 3.5%. The yield of recovered mannogalactan was approximately constant (86%) down to 0.3% borax concentration, below which a significantly greater amount was lost through solubility. About 6 to 7% of the raw material is protein which is soluble in the alkaline medium.

## PRELIMINARY CONVERSIONS OF LOCUST BEAN AND GUAR GUMS

Seventeen grams of borax ($Na_2B_4O_7(10H_2O)$) were dissolved in 500 ml. of water and 50 grams of the gum were added with stirring. Then sufficient sodium hypochlorite solution was added to provide 11 to 12% of available chlorine on the weight of gum. The reaction was allowed to proceed with continuous stirring. Usually a 6° rise in temperature was noted. At the desired time, the mixture was filtered on a Buchner funnel, washed with a small amount of borax solution, and air dried. Variations in this procedure are noted in Table II. The consumption of chlorine was followed by filtering a small sample of the liquid through a fritted glass crucible and titrating the available chlorine with potassium iodide and sodium thiosulphate in the usual manner. These data are given in Table III.

**Table I. Summary of Properties of Locust Bean Gum Treated With Various Concentrations of Borax Solution**

| *Concn. of Borax, %* | *Volume of Hydrated Gum after 24 hr.* | *Yield of product, gram* | *Ash (oven-dry basis), %* | *$H_2O$, %* | *Oven-dry ash-free yield, gram* | *Yield of Mannogalactan, %* |
|---|---|---|---|---|---|---|
| 3.4 | 37 | 4.7 | 7.33 | 15.3 | 3.64 | 86.3 |
| 2.72 | 37 | 4.4 | 6.82 | 11.8 | 3.58 | 84.8 |
| 2.04 | 40 | 4.5 | 5.44 | 10.7 | 3.77 | 89.1 |
| 1.36 | 40 | 4.3 | 4.21 | 10.4 | 3.68 | 87.2 |
| 0.68 | 47 | 4.2 | 2.41 | 8.6 | 3.74 | 88.6 |
| 0.34 | 54 | 4.1 | 1.5 | 9.2 | 3.66 | 86.8 |
| 0.17 | 63 | 3.9 | 0.84 | 9.7 | 3.48 | 82.5 |

The data of Tables II and III are typical of a larger number of conversions made in a similar way. From these experiments several important variables were noted, some of which are explainable but others of which require further investigation.

## EFFECT OF PROTEIN CONTENT ON CONVERSION REACTION

When the hypochlorite and gum were mixed, a very rapid initial reaction occurred during which a con-

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**Table II. Summary of Converting Conditions and Properties of Several Converted Locust Bean and Guar Gums**

| Sample | Borax in medium during conversion, % | Available chlorine on gum,[a] % | Approximate pH of medium | Time of conversion, hr. | Ash, % | Yield (ash-free, oven-dry), % | Relative viscosity 1%, 30°C. | Remarks |
|---|---|---|---|---|---|---|---|---|
| | | | | Locust bean gum | | | | |
| 1 | 2.8 | 12 | 12 | 4 | 10.3 | 82.8 | 261.1 | ..... |
| 2 | 2.8 | 12 | 12 | 16 | 15.2 | 80.8 | 222.1 | ..... |
| 3 | 2.8 | 12 | 12 | 54 | 9.9 | 83.5 | 132.3 | ..... |
| 4 | 1.12 | 12 | 12 | 54 | 9.6 | 82.8 | 5.9 | ..... |
| 5 | 1.12 | 12 | 12 | 72 | 8.7 | 82.5 | 5.2 | ..... |
| 6 | 1.57 | 11 | 14 | 5.5 | 11.2 | .. | 3.9 | Medium 3*N* NaOH |
| 7 | 1.57 | 11 | 7.7 | 2.0 | 8.9 | 79.6 | 6.8 | Buffered at pH 7.7 |
| 8 | 1.57 | 22 | 12 | 6.0 | 7.4 | .. | 1.7 | Two-stage oxidation |
| 9 | 1.57 | 11 | 12 | 5.5 | 6.3 | .. | 15.7 | Had previous alkaline extraction |
| | | | | Guar | | | | |
| 10G4-2 | 1.12 | 12 | 12 | 43 | 5.5 | 76.3 | 9.26 | ..... |
| 11G4-2 | 1.57 | 22 | 12 | 3.0 | 4.5 | .. | 25.7 | Two-stage oxidation |

[a] This was the amount of available chlorine added at the beginning of the reaction.

siderable quantity of gas was liberated. Examination revealed that this gas was principally nitrogen, indicating that a large part of the oxidant was consumed by protein rather than by mucilage. It was shown that about 80% of the available chlorine was consumed during the first half hour of the reaction; the remaining 20% was consumed at a slower rate. Several experiments were made to determine the possible influence of the nitrogen or protein content of the mucilage on the chlorine consumption. Two two-stage conversions were made, one with locust bean gum and the other with guar G4-2. The conversions were started with 11% of available chlorine (as sodium hypochlorite) and after 105 minutes an additional 11% of chlorine was added. The rate of chlorine consumption was determined by titration of the available chlorine at various intervals. These experiments were followed by converting the same gums which had first been extracted with a solution of borax and sodium carbonate to remove some of the protein. The data are given in Tables II and III and are plotted in Fig. 2.

The curves of Fig. 2 indicate that the conversion reaction is quite dependent upon the amount of protein present in the raw mucilage. It required considerably less chlorine to change the rate of oxidation of locust bean gum than to change that for the guar G4-2 which had a somewhat higher protein content and considerable seed coat material. When the raw gums were previously extracted with alkali to remove some of the protein, a much different curve resulted.

**Table III. Comparative Data on the Rate of Chlorine Consumption under Various Experimental Conditions**

| Conversion time, min. | Total available chlorine present, gram | Chlorine consumed, % | Conversion time, min. | Total available chlorine present, gram | Chlorine consumed, % |
|---|---|---|---|---|---|
| Guar No. 11, not alkaline extracted<br>Nitrogen = 1.16%<br>Reaction at pH 12 | | | Locust bean gum No. 8, not alkaline extracted<br>Nitrogen = 1.07%<br>Reaction at pH 12 | | |
| 0 | 5.50 | 0.0 | 0 | 5.6 | 0.0 |
| 15 | 0.75 | 86.4 | 5 | 2.1 | 62.5 |
| 45 | 0.63 | 88.6 | 25 | 1.05 | 81.3 |
| 90 | 0.53 | 90.5 | 60 | 0.75 | 86.6 |
| 105 (added more NaOCl) | 6.03 | 0.0 | 95 | 0.65 | 88.5 |
| 110 | 1.62 | 73.1 | 105 (added more NaOCl) | 6.10 | 0.0 |
| 115 | 1.42 | 76.4 | 115 | 5.14 | 15.7 |
| 135 | 0.90 | 85.2 | 120 | 5.00 | 18.0 |
| 150 | 0.78 | 87.1 | 135 | 4.60 | 24.6 |
| 180 | 0.68 | 88.8 | 190 | 4.11 | 32.6 |
| | | | 360 | 2.70 | 55.8 |
| Guar No. 12, alkaline extracted<br>Nitrogen = 0.37%<br>Reaction at pH 12 | | | Locust bean gum No. 9, alkaline extracted<br>Nitrogen = 0.56%<br>Reaction at pH 12 | | |
| 0 | 5.5 | 0.0 | 0 | 5.55 | 0.0 |
| 20 | 2.8 | 49.1 | 10 | 3.62 | 34.8 |
| 60 | 2.43 | 55.8 | 20 | 3.04 | 45.2 |
| 180 | 1.77 | 67.8 | 45 | 2.60 | 53.2 |
| 330 | 1.28 | 76.7 | 120 | 2.08 | 62.5 |
| 390 | 1.11 | 79.8 | 330 | 1.50 | 73.0 |
| Guar No. 14, alkaline extracted<br>Reaction at pH 12 | | | Locust bean gum No. 7, alkaline extracted<br>Reaction at pH 7.7 | | |
| 0 | 5.5 | 0.0 | 0 | 5.6 | 0.0 |
| 15 | 2.67 | 51.4 | 3 | 3.36 | 40.0 |
| 60 | 2.01 | 63.4 | 14 | 2.28 | 59.3 |
| 100 | 1.55 | 71.8 | 30 | 1.74 | 68.9 |
| 180 | 1.22 | 77.8 | 60 | 1.22 | 78.3 |
| 300 | 0.57 | 89.8 | 120 | 0.84 | 85.0 |
| 360 | 0.40 | 92.8 | | | |

**Table IV. Tub-Size Characteristics of Several Locust Bean and Guar Gum Products Converted in a Borax Medium**

(100% Rag Stock)

| Gum | Relative viscosity at 1%, 30°C. | Tub-size conditions solids at 30°C., % | Basis weight (17 × 22—500), lb. | Bursting strength, pt./100 lb. | Increase in burst, % | MIT fold In | MIT fold Across | Gurley porosity, sec./100 cc. | Elmendorf tear, g./sheet In | Elmendorf tear, g./sheet Across |
|---|---|---|---|---|---|---|---|---|---|---|
| Average blank | .... | ... | 18.7 | 160 | .. | 217 | 57 | 225 | 92 | 102 |
| LBG[a] | 132.0 | 1.0 | 19.2 | 208 | 30.0 | 395 | 83 | 230 | 98 | 110 |
| LBG | 5.2 | 1.0 | 19.6 | 196 | 22.5 | 290 | 80 | 197 | 95 | 116 |
| | 5.2 | 3.5 | 19.5 | 233 | 45.6 | 526 | 142 | 332 | 92 | 105 |
| LBG | 15.7 | 1.0 | 19.0 | 215 | 34.4 | 354 | 88 | 207 | 92 | 103 |
| LBG | 23.8 | 1.0 | 19.1 | 204 | 27.5 | 366 | 88 | 221 | 90 | 101 |
| LBG | 6.8 | 1.0 | 19.0 | 209 | 30.6 | 413 | 93 | 193 | 94 | 106 |
| Guar G44 | 6.8 | 1.0 | 19.0 | 203 | 26.9 | 290 | 76 | 172 | 90 | 104 |
| | 6.8 | 2.0 | 19.0 | 209 | 30.6 | 399 | 76 | 196 | 89 | 99 |
| | 6.8 | 2.5 | 19.1 | 215 | 34.4 | 401 | 102 | 216 | 87 | 98 |
| Guar G44 | 3.7 | 1.0 | 18.7 | 195 | 21.9 | 264 | 75 | 162 | 87 | 97 |
| | 3.7 | 2.0 | 18.9 | 214 | 33.8 | 357 | 102 | 184 | 84 | 94 |
| | 3.7 | 3.5 | 19.1 | 227 | 41.9 | 488 | 100 | 217 | 85 | 92 |
| Starch 1 | 1.63 (2%) | 3.0 | 19.1 | 203 | 26.9 | 379 | 83 | 174 | 92 | 100 |
| Starch 2 | 1.60 (2%) | 5.0 | 20.7 | 205 | 28.1 | 429 | 122 | 249 | 97 | 107 |

[a] Locust bean gum.

It is believed that the reaction of sodium hypochlorite with protein is much more rapid than that with the mannogalactan. Thus, when the reaction begins, both protein and mannogalactan are oxidized but the reaction with protein is so much more rapid that as long as a significant amount of protein remains, the gum, relatively speaking, is only slightly attacked. Only after the protein has been oxidized does the attack occur principally upon the gum itself. Therefore, the impurities in the raw gums certainly play a significant role in the conversion and it is advantageous to start with as pure a product as possible and to remove much of the protein before conversion.

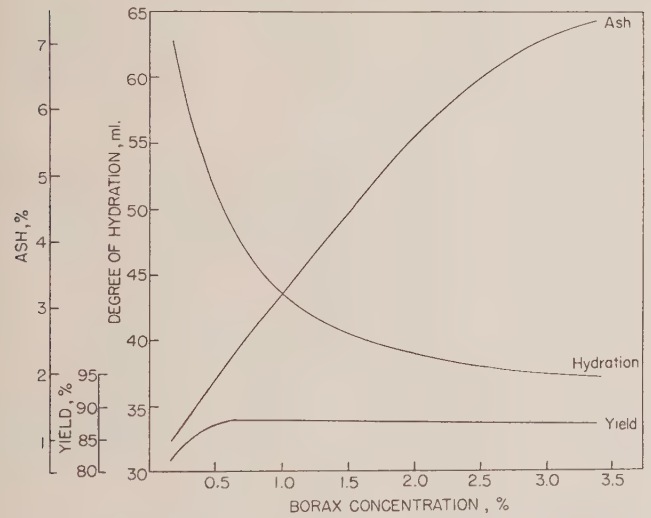


***Fig. 1. Variation of ash, yield, and degree of hydration of locust bean gum at different borax concentrations***

It was found that extraction of the raw mucilage for 0.5 hour with a solution 2% in borax and 0.5% in anhydrous sodium carbonate at 45 to 50°C. gave a product much lower in protein and which still filtered quite rapidly. This method removes about 60% of the protein present. It is apparently impossible to remove all the nitrogen-containing material from the crude gum by alkaline extraction. On the other hand, it is possible that a certain amount of residual protein may be desirable during the conversion to offset the strongly degradative effects which are present during the first part of the reaction when the hypochlorite is more concentrated. However, the lower the protein content prior to conversion, the less chlorine will be required to convert the mannogalactan polymer to a desirable viscosity. Interestingly enough, even the converted products contain appreciable amounts of nitrogen (0.1 to 0.2%). This may or may not be proteinaceous in character.

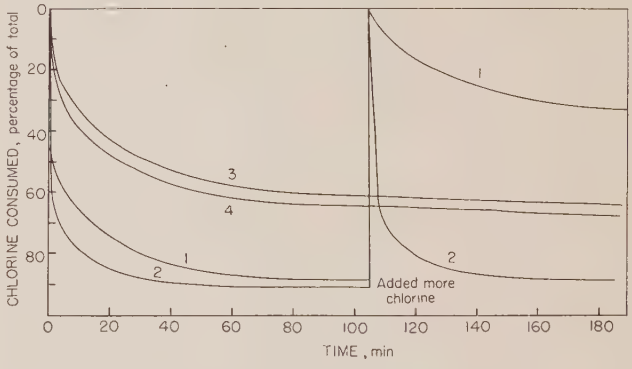


***Fig. 2. Consumption of chlorine by locust bean gum and guar at various nitrogen contents***

1. LBG No. 8 (Nitrogen = 1.07%); 2. Guar No. 11 (Nitrogen = 1.16%); 3. LBG No. 9 (Nitrogen = 0.56%); and 4. Guar No. 12 (Nitrogen = 0.37%).

### GENERAL EFFECT OF ALKALI CONCENTRATION AND pH

Several conversions of both guar and locust bean

**Table V. Data on the Commercial Mill Scale Trial of Converted Guar Mucilage**

(50% Rag Bond Stock)

| Tub-size material | Basis weight (17 × 22—500), lb. | Bursting strength, pt./100 lb. | Increase in burst, % | MIT fold In | MIT fold Across | Elmendorf tear, g./sheet In | Elmendorf tear, g./sheet Across | Valley size, sec. | Gurley porosity, sec./100 cc. |
|---|---|---|---|---|---|---|---|---|---|
| Machine sheet | 16.4 | 120 | .. | 39 | 16 | 51 | 56 | 17 | 59 |
| Starch at 8% | 17.3 | 169 | 40.8 | 165 | 46 | 49 | 52 | 14 | 139 |
| Converted guar at 1.5% | 16.5 | 159 | 32.5 | 101 | 45 | 51 | 58 | 12 | 52 |
| Converted guar at 1.0% | 16.2 | 154 | 28.3 | 76 | 35 | 52 | 55 | 12 | 47 |

NOTE: All sheets conditioned at 50% R.H. and 73°F.

Table VI. Data on Clay Coatings Made with Converted Guar Adhesive

| Type of adhesive | Adhesive on the clay, % | Solids applied to sheet, % | Dennison wax test | K and N ink absorption, average reflectance, % | | |
|---|---|---|---|---|---|---|
| | | | | $R_0$ original | $R_0$ inked | Difference |
| Guar G44 | 10 | 25 | 3A | 75.5 | 43.5 | 32.0 |
| Guar G44 | 12 | 25 | 5A | 75.0 | 44.1 | 30.9 |
| Guar G44 | 12 | 28 | 5A | .. | .. | .. |
| Locust bean gum | 12 | 30 | 5A | .. | .. | .. |
| Starch | 18 | 40 | 4A | 80.1 | 36.6 | 43.5 |
| Starch | 22.5 | 40 | 5A | 78.7 | 39.6 | 39.1 |
| Starch[a] | 22 | 45 | 5A | 70.4 | 43.5 | 26.9 |
| Casein | 12 | 40 | 5A | .. | .. | .. |

[a] Machine finished and calendered; all others not calendered.

gum were carried out at different pH to investigate, in a preliminary way, the effect on the speed of the reaction. It was found that the oxidation is greatly affected by the pH or alkali concentration. Typical data are given in Table III where it is evident that, at pH 7.7, the reaction rate is many times more rapid than at pH 12 and is still more rapid at pH 7.2. Similar results were obtained with guar mucilage. As the alkali concentration is increased—e.g., 3 *N* in NaOH—the reaction rate again speeds up, although it does not approach the speed of pH values in the range of 7.5 to 8.5.

Due care must be exercised during reactions carried out at lower pH values. The mannogalactan must, of course, remain insoluble in the conversion medium and, therefore, the pH must not be too near the neutral point because, at pH 7, solubilization begins immediately. Boric acid has no insolubilizing action on the mucilage. Since sodium hypochlorite solutions characteristically produce HCl during their reaction, the alkalinity decreases and, if insufficient buffer is present, the pH will fall rapidly toward the neutral point. Therefore, when carrying out oxidations at pH values close to the neutral point the mixture must be watched very closely and either a strongly buffered medium must be used or occasional additions of alkali must be made. High alkali concentrations, insofar as investigated, do not cause an appreciable solubilization beyond that occurring at a mildly alkaline pH.

It is probable that the optimum type of conversion would be one in which the initial pH is high enough so that the final pH would be about 7.5. This would enable the first part of the reaction to proceed rapidly under alkaline conditions because of the high concentration of sodium hypochlorite. The latter part of the reaction would also proceed rapidly because of a low pH and the swelling of the mannogalactan. This would give the shortest time of conversion and the highest economy of chlorine. Further work of a fundamental nature has been carried out on purified guar mannogalactan (Haug, A. J., *Tappi* 32, no. 9: 410–413 (Sept., 1949).

## EVALUATION AS TUB-SIZING ADHESIVES

Several of the converted locust bean and guar gums were used as tub-size adhesives on the laboratory scale, employing a 100% rag stock. The tub-size solutions were prepared by cooking the converted gums in water containing enough acid to bring the pH to about 5.5. Low concentrations of tub-sizing solution were used for two reasons—(a) some of the products were too viscous to be used at higher concentrations and (b) outstanding adhesive characteristics are more evident. The data presented in Table IV indicate that these converted gums are outstanding tub-size adhesives.

Since the laboratory trials of converted guar gum indicated good properties, it was decided that a mill trial should be made. A number of 6-pound batches of guar G44 gum were extracted with alkali and converted with sodium hypochlorite in a manner similar to the smaller batches. About 40 pounds of the product were prepared. Trials of a composite sample in the laboratory as a tub size indicated that the product was equal in adhesive properties to the smaller batches.

The converted guar was evaluated under mill conditions, using a 16-pound, 50% rag bond stock. It was found that the sheet was not strong enough to be used with the converted mucilage at 3% concentration and it was necessary to dilute to 1.5 and 1% for the actual run. This was disappointing inasmuch as the mucilage could not be evaluated under the optimum conditions. An 8% starch tub-sizing solution was run on the same sheet for comparison. The machine speed was about 175 f.p.m. and the temperature in the tub was 125°F.

A wet sample was removed after the press and weighed before and after drying. From these data it was found that the starch tub-sized sheet picked up about 4.3% starch; the converted guar at 1.5% picked up 0.72% mucilage and at 1.0%, 0.48% mucilage. The data on the tub-sized sheets are presented in Table V.

The data obtained with the 1.5% solution show the interesting possibilities of converted guar as a tub size. It is believed that a 2.5 to 3.0% solution of the mucilage would have equaled or bettered the 8% solution of starch in strength properties. A slightly greater degree of conversion of the mucilage will make this possible.

## LABORATORY EXPERIMENTS AS A CLAY-COATING ADHESIVE

Several converted products were tested as clay-coating adhesives and compared with starch. The data are presented in Table VI.

The experiments were made at a relatively low coating solids concentration because the mucilages were only moderately converted and possessed viscosities too high for coating at higher solids. The results are encouraging, however, because 12% of the converted mucilage adequately bound the pigment to the sheet, thereby equaling in this property the same amount of casein.

The coatings containing mucilage appear to be somewhat less receptive to printing ink than comparable starch coatings. Further work may result in an improvement in this property.

The converted products contain sufficient borax (boric acid when cooked) to cause a serious viscosity increase in the coating color if the pH is permitted to go to the alkaline side. This should be avoided. However, after the coating mixture has been applied to the sheet, the coating may be made temporarily water insoluble by the addition of a weak alkali which forms the borax gel-mucilage complex.

At the present time converted gums of the mannogalactan type are not commercially available. This is due, primarily, to the limited amounts and relatively high price of raw material. However, it is to be expected that, as soon as this situation becomes more favorable, these converted gums will become interesting commercial products for the paper and allied industries.

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# Further Studies on the Lethal Effect of Drier Roll Temperature upon Bacteria in Paperboard

B. F. SHEMA

**It has been shown that a heat-resistant strain of *Escherichia coli* (Frank) is killed in paperboard during the drying process. The experimental data collected show that the heat of the drier rolls is sufficient to kill all the coliform bacteria present, regardless of their position in the sheet. The total bacterial count of the wet web was reduced by 94.3%.**

The significance of bacterial count as an index of the sanitary quality of paper, especially that which is destined for use in the food packaging industry, has been a controversial issue for the past decade. During this period several attempts have been made to introduce a bacteriological standard which would apply directly to those paper products which come in contact with dairy foods—such as milk, cream, butter, ice cream, cottage cheese, and cheese. As a result of these proposals, a bacteriological standard was accepted by several food control groups. This standard stated that paper or paperboard which is destined for use in the packaging of food, especially milk, shall have a bacterial count of not more than 250 colonies per gram of disintegrated fiber. These standards were proposed and accepted, apparently, without consideration of the number, type, or the sanitary significance of the organisms that were present.

It is common knowledge that many of the procedures used in the manufacture of pulp and paper are lethal to bacteria. These processes are especially germicidal to microorganisms which are significant in the field of public health. In the manufacture of chemical pulp, the wood chips are cooked in either strong acid or alkali. The minimum temperatures attained during a normal cook are 140 to 145°C., and these temperatures are maintained for at least four to five hours, which is certainly a sufficient time to kill all living microorganisms. However, when pulp is manufactured mechanically, extremely high temperatures are not attained but temperatures which are lethal to pathogenic organisms are maintained. Thus, the bacteria surviving this grinding operation are either the heat-resistant and/or the spore-bearing types. At the present time, a considerable portion of the chemically and mechanically produced pulp is bleached with either chlorine or hydrogen peroxide. It is not necessary to elaborate upon the disinfecting power of either of these two effective bleaching agents. In a study of the lethal effect of bleach liquor on the bacterial content of sulphite pulp, it was found that very small additions of chlorine considerably reduced the number of viable bacteria (*1*). However, it was demonstrated that at least one species, *Bacillus luteus*, a nonpathogenic aerobic spore-bearing type, could survive the bleaching operation.

After the wood pulp is prepared, the fibers are suspended in large volumes of water. This fresh or recirculated water is the vehicle which transports this fiber to and through the various processes in the manufacture of paper. This transporting medium contains viable microorganisms. These fungi and bacteria are nonpathogenic types, consisting chiefly of the following species: *Aerobacter aerogenes, Bacillus megatherium, Bacillus mycoides, Bacillus subtilis, Bacillus peptogenes, Achromobacter species,* etc. Fungi in varying numbers are almost always present, including various members of the Saprolegneous group, *Aspergillus sp., Penicillium sp., Mucor sp., Monilia sp., Oidium;* representatives of many other genera can be isolated. These fungi and bacteria are common not only to the pulp and paper industry but they are present also in varying numbers upon or within the various perishable foods that comprise our daily diet. Because of the nature of pulp preparation, it is impossible to avoid completely the introduction of some of the air-borne microorganisms into the pulp, especially during the process of pulp preparation.

In the formation of a sheet of paper, the pulp is either picked up by a woolen felt or formed upon a wire cloth. In either case, the fiber is freed of excess water and formed into a fibrous mat. This wet mat (66% water) is passed over a series of steam-heated drier rolls. The lethal effect of these drier rolls upon bacteria has been adequately demonstrated by several workers (*2, 3, 4*). However, in these experiments, the test organisms were sprayed upon the surface of the sheet and their survival determined. These studies demonstrated conclusively that only nonpathogenic spore-bearing bacteria survive.

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This present investigation is concerned chiefly with the determination of the lethal effect of the drier rolls upon a heat-resistant strain of *Escherichia coli* (Frank strain) when it is present in the board rather than when it is found upon the surface of the sheet.

## EXPERIMENTAL PROCEDURE

A six-cylinder board machine was selected for this study. This particular machine produced a paperboard, the furnish of which was composed of all grades of waste paper. This type of mill, the type of machine, and the source of raw stock were selected for two reasons: first, this type of furnish has the greatest number and a very diversified microflora and, second, it has the greatest protective effect upon bacteria, especially those which are important in public health.

In this study, the wet web was inoculated immediately after the felt picked up the fiber from the cylinder mold. Thus, in this manner, it was possible to inoculate the various layers of the board immediately after they were formed. The concentrated suspension of the heat-resistant strain of *Escherichia coli* was sprayed uniformly on the wet web with a Tornado electric sprayer. A long, relatively narrow strip was sprayed with the bacterial suspension. A portion of this marked strip was sampled at the wet end of the machine and another sample was obtained at the dry end of the machine. The wet sample was collected just before the wet web entered the press section. Preliminary experimental data showed that the press section did not exert a marked effect upon the bacterial content of this type of paperboard. A sample of a comparable sprayed area was obtained after the board passed through the drier section, but before the board entered the calender. This procedure was followed for each of the six cylinder molds. A control was included for each of the experimental runs.

In this study, the survival of *Escherichia coli* was determined in boards of three different caliper—namely, 0.016, 0.023, and 0.030 inch. Each experimental trial for the three different board weights was replicated four times. The replicates were run on separate days, and were not necessarily in consecutive order.

The wet web samples were collected with sterile forceps and placed in sterile sample bottles. The dry paperboard was cut and aseptically placed into a sterile kraft envelope. In this manner, the samples were transported to the laboratory for analysis of total bacterial count and the number of coliform bacteria. The elapsed time from the actual collection of the sample and the plating out of the sample never exceeded 1.5 hours. The wet and dry samples were plated in the manner prescribed for the bacteriological examination of paper and paperboard (*5*). The number of coliform bacteria was determined by plating out 0.1 gram of the disintegrated fiber in Violet red bile agar. A 0.1-gram sample of the disintegrated fiber was also placed into a tube of Brilliant green bile and into a tube of formate ricinoleate broth. The experimental results have shown that more consistent results were obtained with the formate ricinoleate broth than with the use of Brilliant green bile, because the latter medium gave many false positives. In any event, when a tube of broth was positive, it was confirmed by streaking a culture upon the surface of either Endo's or Levine's eosin Methylene blue agar.

During the course of this investigation, the temperature of the drier rolls and the temperature of the surface of the sheet were determined as the paper passed through the bank of 80 drier rolls. The temperature of the drier rolls did not vary appreciably, even though a different thickness of board was passing over them. In general, however, the temperature of the first drier roll was somewhat lower than that of the last drier roll. The extreme variation in temperature for the 80 drier rolls never exceeded 50°F. The temperature upon the surface of the sheet was relatively low (90°F.) during the first stages of drying because of the rapid loss of water. However, this rapid cooling of the drier rolls was overcome after the paperboard passed over the sixth drier roll. At this point the temperature of the surface of the sheet was at least 120° F. and increased to 190 to 200°F. as the paperboard passed over the eighth drier roll. The temperatures of the drier rolls and the temperature of the surface of the sheet were obtained by the use of a hand model Alnor Tyrocon pyrometer. The pressure of the steam which furnished the heat to the drier rolls was relatively uniform for all calipers studied. The normal range of this steam pressure was 15.5 to 16 p.s.i. The drying time did not vary appreciably during the production of a board of any one caliper. The average drying time for the 0.016-caliper board was 3 minutes and 45 seconds; for the 0.023-caliper board, 5 minutes and 20 seconds, and for the 0.030-caliper board, 6 minutes and 2 seconds. Under these conditions of drying, the board is held at a higher temperature for a longer period of time than those which are recommended for the complete pasteurization of milk.

**Table I. Average Bacterial Content of the Wet Web and the Finished Paper for 0.016-Caliper Board**

| Sample | Average bacterial count, number colonies per gram | | | | | |
|---|---|---|---|---|---|---|
| | Wet Web[a] | | Paperboard[b] | | | |
| | Total count | Coliform | Total Count | Kill, % | Coliform | Kill, % |
| Uninoculated control | 236,800,000 | 215,000 | 382,000 | 98.4 | 0 | 100 |
| *E. coli* inoculated after vat 1 | 193,000,000 | 1,196,000 | 404,000 | 97.9 | 0 | 100 |
| *E. coli* inoculated after vat 2 | 231,000,000 | 810,000 | 373,000 | 98.4 | 0 | 100 |
| *E. coli* inoculated after vat 3 | 224,600,000 | 1,338,000 | 216,000 | 99.0 | 0 | 100 |
| *E. coli* inoculated after vat 4 | 222,200,000 | 632,000 | 432,000 | 98.1 | 0 | 100 |
| *E. coli* inoculated after vat 5 | 242,600,000 | 485,200 | 466,000 | 98.0 | 0 | 100 |
| *E. coli* inoculated after vat 6 | 252,600,000 | 680,000 | 450,000 | 98.2 | 0 | 100 |

[a] 33% fiber.
[b] 97% fiber.

## DISCUSSION AND EXPERIMENTAL RESULTS

The sterilizing effect of the drier rolls is generally at-

tributed to the action of moist heat upon bacteria. It has been shown that only the spore-bearing bacteria survive in the finished paper. The experimental results show that the heat-resistant strain of *E. coli* does not survive the lethal effect of the drier rolls. The data presented in Tables I, II, and III give the total bacterial count and the number of coliform bacteria in the wet web and in the finished paperboard.

The data for the 0.016-caliper board are presented in Table I. From these data it is evident that the initial bacterial content of the stock was very high. The total bacterial count of the wet web was generally in the hundreds of millions of bacteria per gram, whereas the number of coliform bacteria were considerably less, even when the organism was sprayed upon the wet web. The coliform count for the wet web ranged from 215,000 colonies per gram for the control to 1,338,000 colonies per gram for one of the inoculated samples. In general, the inoculated samples had a much higher coliform count than the uninoculated control. The data show that great numbers of bacteria were killed during the drying process. Bacterial counts which were in the hundreds of millions in the wet web were reduced to hundreds of thousands in the finished paper, a kill of at least 98%. In the case of the coliform bacteria, all were killed during the drying process. The average kill for all species of bacteria was 98.3% for the 0.016-caliper board.

The data for the 0.023-caliper board are presented in Table II. The bacterial content of the wet web for this board was considerably lower than for either the 0.016- or 0.030-caliper board. However, the number of bacteria surviving the drier rolls is about the same. Therefore, it can be concluded that about the same number of spore-bearing bacteria were present in this board as were present in either the 0.016- or 0.030-caliper board. Because of this rather consistent number of spore-bearing bacteria and the lower bacterial count of the wet web, it would appear that the drier rolls were not as effective when 0.023-caliper board was manufactured. On a percentage basis, the total bacterial content was reduced by 93.7% and, again, all the coliform bacteria were killed.

The data for the 0.030-caliper board are presented in Table III. It is again evident that the bacterial count of the wet web was very high, as was also the number of coliform bacteria. Again, the drier rolls killed all the coliform bacteria and reduced the total bacterial content by at least 94.9%.

**Table II. Average Bacterial Content of the Wet Web and the Finished Paper for 0.023-Caliper Board**

| Sample | Average bacterial count, number colonies per gram | | | | |
|---|---|---|---|---|---|
| | Wet Web[a] | | Paperboard[b] | | |
| | Total count | Coliform | Total Count | Kill, % | Coliform | Kill, % |
| Uninoculated control | 82,440,000 | 197,358 | 508,000 | 93.8 | 0 | 100 |
| *E. coli* inoculated after vat 1 | 98,660,000 | 507,200 | 602,500 | 93.8 | 0 | 100 |
| *E. coli* inoculated after vat 2 | 76,200,000 | 573,500 | 582,000 | 92.4 | 0 | 100 |
| *E. coli* inoculated after vat 3 | 83,580,000 | 856,600 | 576,000 | 93.2 | 0 | 100 |
| *E. coli* inoculated after vat 4 | 84,780,000 | 1,256,000 | 624,000 | 92.7 | 0 | 100 |
| *E. coli* inoculated after vat 5 | 89,520,000 | 499,034 | 391,600 | 95.7 | 0 | 100 |
| *E. coli* inoculated after vat 6 | 96,460,000 | 374,400 | 550,000 | 94.3 | 0 | 100 |

[a] 33% fiber.
[b] 97% fiber.

The data presented in this report show that coliform bacteria do not survive the lethal effect of drier roll temperature. The data demonstrate, further, that the position of these bacteria in the sheet is not important—that is, *E. coli* is killed whether it is on the surface or in the center of the board. If coliform bacteria in and on the sheet are killed on the driers, it follows that other bacteria of public health significance would also be destroyed. The bacteria that remain would be nonpathogenic spore-forming varieties.

**Table III. Average Bacterial Content of the Wet Web and the Finished Paper for 0.030-Caliper Board**

| Sample | Average bacterial count, number colonies per gram | | | | |
|---|---|---|---|---|---|
| | Wet Web[a] | | Paperboard[b] | | |
| | Total count | Coliform | Total count | Kill, % | Coliform | Kill, % |
| Uninoculated control | 275,000,000 | 254,400 | 1,379,000 | 95.0 | 0 | 100 |
| *E. coli* inoculated after vat 1 | 231,600,000 | 641,000 | 1,413,000 | 93.9 | 0 | 100 |
| *E. coli* inoculated after vat 2 | 228,500,000 | 1,124,500 | 1,449,000 | 93.7 | 0 | 100 |
| *E. coli* inoculated after vat 3 | 235,600,000 | 1,588,000 | 1,106,000 | 95.3 | 0 | 100 |
| *E. coli* inoculated after vat 4 | 235,400,000 | 4,577,000 | 1,015,000 | 95.7 | 0 | 100 |
| *E. coli* inoculated after vat 5 | 238,600,000 | 1,218,000 | 1,102,000 | 95.4 | 0 | 100 |
| *E. coli* inoculated after vat 6 | 254,000,000 | 720,000 | 1,162,000 | 95.4 | 0 | 100 |

[a] 33% moisture.
[b] 97% moisture.

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# The Hydrolysis of Sodium Sulphide in Mixtures with Sodium Hydroxide

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**A quantitative method has been developed for determining the hydrolysis of sodium sulphide in mixtures with sodium hydroxide over the ranges of concentration and temperature encountered in sulphate pulping. The method is based upon the quantitative solubility of mercuric sulphide in sodium sulphide and its insolubility in sodium hydrosulphide, sodium hydroxide, and water. This method indicated that the sodium sulphide was about 40% hydrolyzed at the beginning of a typical sulphate cook and about 90% hydrolyzed at the end. The concentration of sodium hydroxide in the cooking liquor appeared to be the dominant factor in determining the degree of hydrolysis of the sodium sulphide; temperature and sodium sulphide concentration exerted only a minor influence. The determination of hydrolysis by conductance measurements, pH measurements and, indirectly, by calculation from acidic ionization constants gave only approximate results. These methods had serious limitations as to concentration and/or temperature, although the order of magnitude of the hydrolysis results obtained at 25°C. was such as to lend additional support to the validity of the mercuric sulphide method.**

Our present knowledge of the chemical reactions occuring in a sulphate cook is far from complete. The heterogeneity of these reactions is one of the greatest obstacles preventing their exact definition, but another factor is the lack of information concerning the state of the simple inorganic compounds in the liquor under the conditions of the reaction.

Perhaps the most important consideration in an interpretation of sulphate cooking is the explanation of the role played by the sodium sulphide. Upon hydrolysis, this salt forms sodium hydroxide and sodium hydrosulphide:

$$Na_2S + HOH \rightarrow NaOH + NaHS$$

In order to explain the pulping action of sodium sulphide, some estimation must be made as to the extent of this hydrolysis. The sodium hydrosulphide and the sodium hydroxide formed by the hydrolysis are important participants in the delignification of wood.

It was desired to determine quantitatively the position of this hydrolysis equilibrium for the range of conditions normally encountered in sulphate pulping. This information, coupled with analytical data on the sodium hydroxide and total sulphide contents of the liquor at various points during the cook, would make available the total concentrations of sodium hydroxide, sodium hydrosulphide, and unhydrolyzed sodium sulphide existing in a sulphate cooking liquor throughout a normal sulphate cook.

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There is also considerable sodium carbonate in a typical sulphate cooking liquor; it undergoes a similar hydrolysis to sodium hydroxide and sodium bicarbonate. Some work was done on the carbonate hydrolysis, although sodium carbonate is generally recognized to be a very poor pulping agent and of minor importance in a sulphate cook.

## DETERMINATION OF HYDROLYSIS BY CONDUCTANCE MEASUREMENTS AND BY pH MEASUREMENTS

The determination of hydrolysis by conductivity measurements appeared to be a very promising method for this study since, with relatively simple apparatus, conductance readings have been made at temperatures far in excess of those encountered in sulphate pulping. The conductance of sodium hydroxide is much greater than that of an equal concentration of either sodium sulphide or sodium carbonate. The basis of the conductance method for determining these hydrolyses is the increase in the conductivity of the solution that results when the salt hydrolyzes to form sodium hydroxide and either sodium hydrosulphide or sodium bicarbonate.

The determination of hydroxyl ion concentration through the measurement of the pH of the solution is a conventional method for determining the position of a hydrolytic equilibrium in which a strong base appears as one of the products. From the hydrogen ion concentration, the concentration of hydroxyl ion may be calculated, and the concentration of sodium hydroxide formed by hydrolysis is equal to that of the sodium hydrosulphide formed.

It is also possible to calculate the extent of the hydrolysis from $K_w$, the ion product constant of water, and $K_a$, the ionization constant of the acid of the salt, since the hydrolysis constant ($K_h$) is eqaul to $K_w/K_a$. Values for $K_w$ and $K_a$ are available in the literature.

Table I presents hydrolysis values for sodium sulphide solutions at 25°C., as determined by the methods described above. Values for sodium carbonate solutions at 25°C. appear in Table II. The degree of

**Table I. Hydrolysis of Sodium Sulphide (25°C.)**

| *Molarity of $Na_2S$* | *Hydrolysis from conductance, %* | *Molarity of $Na_2S$* | *Hydrolysis from pH, %* | *Molarity of $Na_2S$* | *Hydrolysis from $K_w/K_a$, %* |
|---|---|---|---|---|---|
| 0.0063 | 73.8 | 0.0050 | 62 | 0.0062 | 92 |
| 0.0126 | 81.8 | 0.0100 | 63 | 0.0123 | 87 |
| 0.0253 | 80.3 | 0.0250 | 63 | 0.0246 | 80 |
| 0.0505 | 79.3 | 0.0500 | 72 | 0.0493 | 69 |
| 0.1010 | 79.2 | 0.1000 | 76 | 0.0985 | 58 |
| 0.202 | 76.3 | 0.200 | 79 | 0.197 | 45 |
| 0.404 | 77.7 | 0.400 | 38 | 0.394 | 35 |

hydrolysis was calculated from conductance data according to Schaeffer's method, as described by Knox (*1*). Schaeffer assumed that the conductance of unhydrolyzed sodium sulphide would be approximately the same as that of sodium hydrosulphide of the same

**Table II. Hydrolysis of Sodium Carbonate (25°C.)**

| *Molarity of $Na_2CO_3$* | *Hydrolysis from conductance, %* | *Hydrolysis from pH, %* | *Hydrolysis from $K_w/K_a$, %* |
|---|---|---|---|
| 0.005 | .... | 11.4 | 16.2 |
| 0.010 | 58.2 | 6.7 | 12.0 |
| 0.025 | 49.8 | 3.6 | 7.5 |
| 0.050 | 44.3 | 2.4 | 5.2 |
| 0.075 | .... | 2.0 | 4.2 |
| 0.100 | 37.5 | 1.7 | 3.6 |
| 0.200 | .... | 1.3 | 2.5 |
| 0.300 | 24.9 | ... | ... |
| 0.400 | .... | 1.1 | 1.7 |
| 0.500 | 19.0 | ... | ... |

molarity. The method of Bredig (*2*) for the calculation of conductance data was found to be inapplicable to the sodium sulphide hydrolysis. Table III gives the hydrolysis of sodium sulphide in mixtures with sodium hydroxide at 25°C., as determined from conductance data.

**Table III. Hydrolysis of Sodium Sulphide in Mixtures with Sodium Hydroxide (25°C.)**

| *Molarity of $Na_2S$* | *Molarity of NaOH* | | | |
|---|---|---|---|---|
| | *0, %* | *0.1, %* | *0.2, %* | *0.4, %* |
| 0.050 | 80 | 69 | .. | .. |
| 0.100 | 78 | 72 | 68 | .. |
| 0.200 | 77 | 76 | 72 | 69 |

Hydrolysis was calculated from pH data according to the following equations:

$$a_{OH^-} = \frac{K_w}{a_{H^+}} = \frac{\gamma_w^2 m_H m_{OH}}{a_{H^+}}$$

$a_{OH^-}$ = activity of the hydroxyl ion
$a_{H^+}$ = activity of the hydrogen ion
$K_w$ = ion product constant for water
$\gamma_w^2$ = ionic activity coefficient product for water
$m_H$ = concentration of hydrogen ion in pure water
$m_{OH}$ = concentration of hydroxyl ion in pure water

$$\frac{h^2}{1-h} = \frac{(a_{OH^-})^2}{[\gamma(c) - a_{OH^-}]\gamma(c)}$$

where

$h$ = degree of hydrolysis of the salt
$c$ = molarity of the salt
$\gamma$ = activity coefficient for the salt anion (first dissociation)

Hydrolysis was calculated from the second ionization constants of carbonic acid and hydrogen sulphide from the following equation:

$$\frac{K_w}{K_2} = \frac{\gamma_1^2(c)h^2}{\gamma_2(1-h)}$$

where

$K_w$ = ionization product for water
$K_2$ = second ionization constant of the acid
$h$ = degree of hydrolysis of the salt
$c$ = molarity of the salt
$\gamma_1$ = activity coefficient for the univalent ion of the salt
$\gamma_2$ = activity coefficient for the bivalent ion of the salt

Activity data suitable for these calculations are difficult to find in the literature, although a very good compilation is given by Robinson and Harned (*3*).

An examination of these data indicates that the hydrolysis values obtained by the three methods are not in agreement and that the data are somewhat erratic.

The established theories relating conductance to other properties of the solution, such as temperature and concentration, hold strictly only at infinite dilution. The application of these theories to the determination of hydrolysis at practical concentrations is a long and arduous procedure, in which careful consideration must be given to various effects arising from ionic and, in the case of weak electrolytes, molecular attraction.

The determination of hydrolysis through pH measurements has a limited application. The upper limit of measurement is a pH of 13, even when the Beckman Type E electrode (a special electrode for high alkalinities made with sodium-free glass) is used. The pH of concentrated sodium sulphide solutions is in this region, before the addition of any sodium hydroxide. Thus, the method is not applicable to any solution with an alkalinity approaching that of a sulphate white liquor. A second limitation is that of temperature, the upper limit for the Type E electrode being 40°C. Finally, the accurate calculation of hydrolysis from pH requires the use of activity data for the hydroxyl ion and the ionic activity coefficient product for water.

The calculation of hydrolysis from the ionization product for water and the second ionization constant of either carbonic acid or hydrogen sulphide is only an approximate method. Once again, data on the ionic activity coefficient product for water and the activities of several of the ions participating in the hydrolytic equilibrium are required. In addition, the second ionization constants of carbonic acid and hydrogen sulphide are not known with the requisite accuracy. The method is not applicable to mixtures of sodium carbonate or sodium sulphide and sodium hydroxide, since the $K_h = K_w/K_a$ relationship does not hold under these conditions. No calculations at elevated temperatures are possible because of the lack of the required activity and ionization data.

In summation, it may be said that the determination of hydrolysis by conductance measurements, pH measurements and, indirectly, by calculation from acidic ionization constants gave only approximate results. These methods have serious limitations as to concentration and/or temperature, although the results obtained at 25°C. indicated that sodium sulphide is a more highly hydrolyzed salt than sodium carbonate.

In the course of the determinations of the degrees of hydrolysis of sodium sulphide and sodium carbonate by conductance methods, data for conductance vs. concentration curves were obtained for the various compounds participating in the hydrolytic equilibria. These data were taken only for use in hydrolysis calculations and, consequently, were not determined with the accuracy and precision that is possible when the most sensitive equipment is used under rigidly standardized conditions. However, since conductance data for several of the compounds are not available in the literature, some of the values are presented in

Table IV. In this work an industrial model conductance bridge (Model RC-1 bridge of Industrial Instruments, Inc., modified to use a 1000-cycle external oscillator) was used, temperature was maintained at 25±0.1°C., conductivity water was employed, and ordinary quantitative techniques were used in the preparation and handling of the solutions. Equivalent conductances at infinite dilution are given, as determined from conductances at various concentrations according to the methods of Kohlrausch and Shedlovsky.

**Table IV. Equivalent Conductances at Infinite Dilution (25°C.) (ohm$^{-1}$cm.$^{-1}$/equivalent weight)**

| *Compound* | *Kohlrausch method* | *Shedlovsky method* |
|---|---|---|
| NaOH | 234.6 | 239.2 |
| $NaHCO_3$ (unhydrolyzed) | 102.3 | 102.8 |
| $NaHCO_3$ | 165.3 | 160.4 |
| $^1/_2Na_2CO_3$ | 130.9 | 127.7 |
| NaHS (unhydrolyzed) | 104.3 | 106.9 |
| $^1/_2Na_2S$ | 131.1 | 138.8 |

## DETERMINATION OF HYDROLYSIS BY THE MERCURIC SULPHIDE SOLUBILITY METHOD

The results obtained by the conductance and pH methods lacked the desired accuracy, and an extensive search was made for other methods of measuring hydrolytic equilibrium, particularly that of sodium sulphide.

Knox (*1*), in 1908, published some very interesting work on the hydrolysis of sodium sulphide at room temperature. He determined the hydrolysis by a method based upon the solubility of mercuric sulphide in sodium sulphide and its insolubility in sodium hydrosulphide, sodium hydroxide, and water. The mercuric sulphide apparently goes into solution by forming a soluble complex. Electromotive force measurements indicate that the complex formed is $Na_2HgS_2$ and, thus, that one mole of mercuric sulphide goes into solution for every mole of sodium sulphide present in the unhydrolyzed form:

$$HgS + Na_2S \longrightarrow Na_2HgS_2$$

Solubility is determined after prolonged mixing of an excess of mercuric sulphide with the solution in question. Finally, the undissolved mercuric sulphide is allowed to settle and an aliquot of the clear, supernatant liquid is pipetted off and analyzed for mercuric sulphide.

## APPARATUS FOR HYDROLYSIS DETERMINATIONS AT 25°C.

For the work at 25°C., the solutions under investigation and an excess of mercuric sulphide were placed in 60-ml. glass-stoppered Pyrex bottles which were rotated at about 5 r.p.m. in a large water bath. Twenty-four hours of mixing were found to be sufficient for reaching equilibrium. The bottles were then allowed to stand in the water bath for at least an hour to allow the excess mercuric sulphide to settle. A 10-ml. portion of the clear, supernatant liquid was then removed for analysis.

## APPARATUS FOR HIGH-TEMPERATURE HYDROLYSIS DETERMINATIONS

For the purpose of determining the position of the sodium sulphide hydrolysis at sulphate cooking temperatures, equipment was designed and constructed to permit investigation of the mercuric sulphide method at elevated temperatures. The apparatus in its final form is shown in Fig. 1.

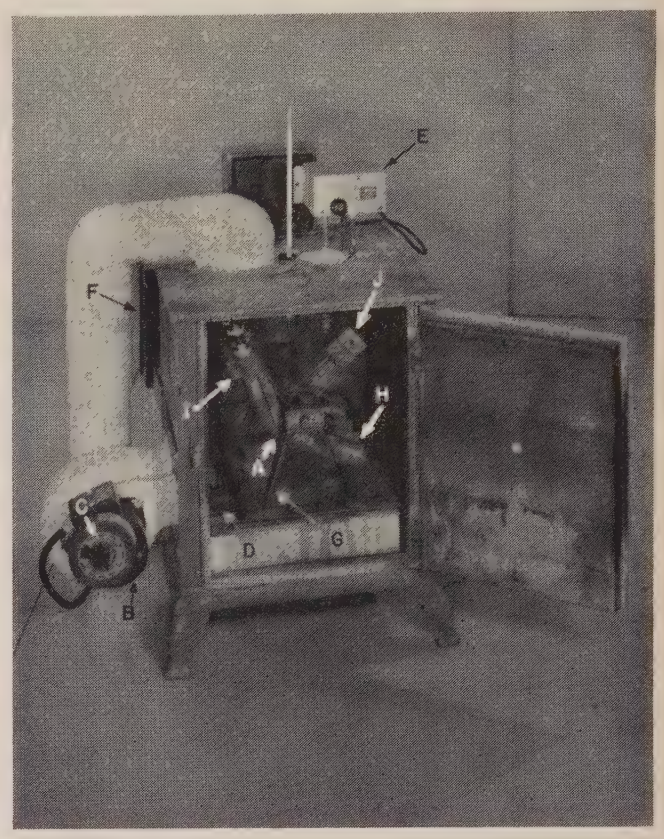


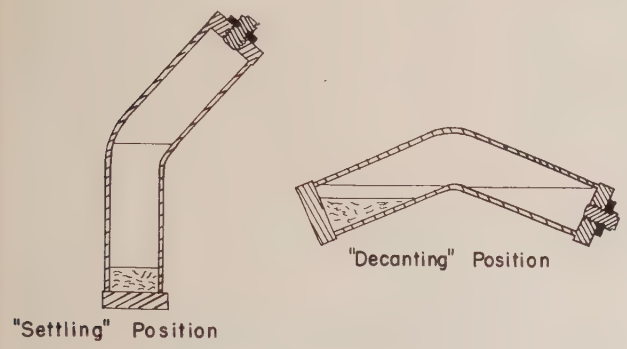

***Fig. 1. Apparatus for high-temperature hydrolysis determinations***

(A) Stainless steel tube, (B) blower motor, (C) blower motor speed control, (D) strip heater, (E) thermoregulator, (F) a.c.-d.c. relay, (G) removable cradle, (H) trough, (I) steel clip, and (J) counterbalance.

High-temperature solubility determinations were carried out in sealed tubes (*A* in Fig. 1) bent at the center to an angle of about 130°. The tubes were made from $^3/_4$-inch stainless-steel tubing with end pieces of solid stainless steel, $^3/_8$ inch thick, silver-soldered to the tubing. One end piece was drilled and tapped for a stainless-steel plug. Dow-Corning silicone rubber was found to be an excellent gasket material giving a pressure-tight seal without excessive tightening of the plug. The tubes, about half filled with the solution under investigation and containing an excess of mercuric sulphide, were rotated end-over-end at about 10 r.p.m. for 24 hours in an oven at 110 or 165°C., after which the excess mercuric sulphide was allowed to settle out at one end of the tube. After settling was complete, the slight bend in the center permitted the decantation of some of the clear supernatant liquid. The tube was then allowed to cool and the separated supernatant liquid was removed at room temperature. An aliquot of the liquid was analyzed for mercuric sulphide. Figure 2 shows cross-sectional views of a tube in the "settling" and "decanting" positions, and indicates the method for the isola-

tion of a sample of supernatant liquid in the right-hand end of the tube by decantation.

The oven for the determinations was equipped with a blower system (*B* and *C*) arranged to take air from the top of the oven and return it along the bottom. The heating element of the oven was rated at 700 watts; an auxiliary 150-watt strip heater (*D*) was necessary to maintain the temperature at 165°C. Temperature control within 0.5°C. was maintained by means of a Cenco-DeKhotinsky Thermo-Regulator (*E*) and an a.c.-d.c. relay (*F*) in series with the main heating element of the oven. The thermometer used was compared with a thermometer calibrated at the Bureau of Standards.



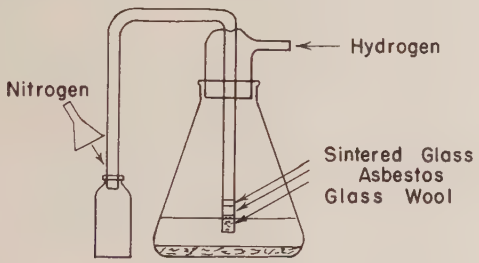




***Fig. 2. (top) Cross section—stainless steel tubes for high-temperature hydrolysis determination. Fig. 3. (bottom) Apparatus for filtration at 100°C.***

A removable cradle (*G*) was constructed for rotation of the tubes. This device consisted of five troughs (*H*), made of $^1/_2$-inch conduit, brazed to a $^5/_{16}$-inch steel shaft. The tubes were held in the troughs by thin steel clips (*I*). The shaft rotated in sleeve-type brass bearings and was coupled through a reduction gear to a small a.c.-d.c. motor mounted to the back of the oven. Dow-Corning stopcock grease (a silicone preparation) was found to be a fairly good bearing lubricant at 165°C. A 500-ohm, 0.31-ampere potentiometer in series with the motor controlled the speed of rotation. An adjustable counterbalance (*J*) was brazed onto the shaft in order to provide a more balanced load for the motor.

The shaft was marked where it extended out of the oven at the rear, so that rotation could be stopped with the tubes in "settling" position and in "decanting" position after settling was complete, without opening the oven.

## ANALYSIS OF THE SUPERNATANT LIQUID FOR DISSOLVED MERCURIC SULPHIDE

Ten milliliters of the clear, supernatant liquid were carefully pipetted into a 50-ml. Erlenmeyer flask containing 10 ml. of 6 *N* hydrochloric acid. The transfer was made under a protective stream of nitrogen in order to minimize atmospheric oxidation. Hydrogen sulphide was then bubbled through the solution for ten minutes to complete the precipitation. The solution was filtered through a weighed Pyrex "fine" sintered-glass crucible to which an asbestos mat had been added. The precipitate was washed with 30 ml. of 95% ethyl alcohol, followed by an equal amount of carbon disulphide. The crucibles were dried for at least 2 hours at 110°C., cooled in a desiccator for 30 minutes, and weighed.

## CALCULATION OF DEGREE OF HYDROLYSIS FROM MERCURIC SULPHIDE SOLUBILITY

The method of calculation is illustrated by the following example: 10 ml. of the supernatant liquid from a solution 0.25 *M* in sodium sulphide and 0.25 *M* in sodium hydroxide, after mixing 24 hours with an excess of mercuric sulphide, contained 0.1173 gram of dissolved mercuric sulphide.

$$\text{Molarity of HgS in solution} = \frac{\text{wt. HgS}}{\text{HgS}} \times \frac{1000}{10}$$

$$= \frac{0.1173}{232.67} \times 100$$

$$M \text{ HgS} = 0.0504$$

Since, in the presence of a large excess of HgS,

$$\text{HgS} + \text{Na}_2\text{S} \longrightarrow \text{Na}_2\text{HgS}_2$$

$$\text{molarity HgS} = \text{molarity Na}_2\text{S as Na}_2\text{S}$$

$$M \text{ Na}_2\text{S} = 0.0504$$

$$\text{Hydrolysis of Na}_2\text{S} = 100 - \frac{0.0504 \times 100}{0.25}$$

$$= 79.8\%$$

## INVESTIGATION OF THE MERCURIC SULPHIDE SOLUBILITY METHOD

The basis for the mercuric sulphide method was tested in the following ways:

### *Mercuric Sulphide Solubility*

The method is dependent upon the insolubility of mercuric sulphide in sodium hydrosulphide, sodium hydroxide, and water. Determinations at 25°C. indicated that the solubilities were negligible at this temperature. At 165°C., some solubility was noted for sodium hydroxide, although the amount was negligible at 110°C. Determinations were made at a number of sodium hydroxide concentrations at 165°C. in order to make possible the application of a correction in the hydrolysis calculations at that temperature. In no case did the correction change the calculated percentage of hydrolysis to an extent greater than 2%.

### *Quantitative Nature of the Mercuric Sulphide-Sodium Sulphide Complex Formation*

The next point of the Knox method to be investigated was the mole for mole solubility of mercuric sulphide in sodium sulphide. The basis for the calculation of the hydrolysis is the proposition that, in the presence of an excess of red mercuric sulphide, there is a quantitative 1:1 relationship between the moles of dissolved mercuric sulphide and the moles of sodium sulphide present in the unhydrolyzed form. Table V shows the hydrolysis values obtained for solu-

tions in which the ratio of sodium hydroxide to sodium sulphide concentration was very high. Very low degrees of hydrolysis were obtained. In run 4, for example, 1.3% hydrolysis was obtained, indicating that the amount of mercuric sulphide dissolved was 98.7% of the total concentration of sodium sulphide (hydrolyzed and unhydrolyzed). With the large excesses of sodium hydroxide present in these solutions, a very low degree of hydrolysis would be expected, although values of a few per cent certainly seem possible. In any event, one may consider the data of Table V as very strong evidence that the mole for mole solubility relationship is quantitative or very nearly quantitative at 25°C. Unfortunately, the appreciable solubility of mercuric sulphide in concentrated sodium hydroxide makes it impossible to obtain evidence of this sort at 165°C.

**Table V. Hydrolysis of Sodium Sulphide in Concentrated Sodium Hydroxide (25°C.)**

| *Run* | *Molarity of $Na_2S$* | *Molarity of NaOH* | *Molarity of HgS in solution* | *Hydrolysis, %* |
|---|---|---|---|---|
| 1 | 0.0483 | 9.31 | 0.0468 | 3.0 |
| 2 | 0.0483 | 9.31 | 0.0466 | 3.4 |
| 3 | 0.1259 | 8.66 | 0.1217 | 3.3 |
| 4 | 0.1259 | 8.66 | 0.1242 | 1.3 |

***Reversibility of the Complex-Formation Reaction***

The reversibility of the reaction of HgS with $Na_2S$ to form a soluble complex was tested in the following manner: a solution 0.3 *M* in sodium sulphide and 1 *M* in sodium hydroxide was rotated with an excess of mercuric sulphide at 25°C. for 24 hours. A sample of the supernatant liquid was analyzed at this time. Sufficient water was then added to make the solution 0.15 *M* in sodium sulphide and 0.5 *M* in sodium hydroxide. After an additional 24 hours of mixing, the supernatant liquid was again analyzed. The results are shown in Table VI.

**Table VI. Reversibility of Mercuric Sulphide-Sodium Sulphide Complex Formation**

| | *M HgS in solution* | *Hydrolysis (25°C.), %* |
|---|---|---|
| 0.3 *M* $Na_2S$, 1 *M* NaOH | 0.1443 | 51.9 |
| After dilution and 24 hr. mixing, 0.15 *M* $Na_2S$, 0.5 *M* NaOH | 0.0500 | 66.7 |

The reversibility of the complex-formation reaction,

$$HgS + Na_2S \rightleftharpoons Na_2HgS_2$$

is a requisite of the high-temperature adaptation of the Knox hydrolysis determination. The reaction tubes are filled at room temperature with the solution in question and an excess of mercuric sulphide, and are then heated to the desired temperature. In order that the method may yield correct results at the elevated temperature, the soluble complex must be able to break down with the resultant precipitation of mercuric sulphide from solution, if the hydrolysis of the sodium sulphide increases. After 24 hours of mixing with an excess of mercuric sulphide at 25°C., the hydrolysis of the sodium sulphide in a solution 0.3 *M* in sulphide and 1.0 *M* in sodium hydroxide was determined as 51.9%, or 0.1443 *M* mercuric sulphide in solution. After the addition of sufficient distilled water to make the solution 0.15 *M* in sodium sulphide and 0.5 *M* in sodium hydroxide with 24 additional hours of mixing, the hydrolysis was determined as 66.7%, or 0.0500 *M* mercuric sulphide in solution. It is evident that considerable mercuric sulphide was thrown out of solution by the increase in hydrolysis upon dilution. In addition, both of the hydrolysis values appear very reasonable when compared with the data at other concentrations in Table IX. The complex-formation reaction appears to be reversible.

***Effect of Complex Formation on the Sodium Sulphide Hydrolysis Equilibrium***

In the development of any method for the determination of the position of an equilibrium, the possibility of disturbing the equilibrium must be carefully considered. In the Knox method, the sodium sulphide in solution participates in two equilibrium reactions.

$$\begin{array}{c} HgS \\ + \\ HOH + Na_2S \rightleftharpoons NaOH + NaHS \\ \downarrow\uparrow \\ Na_2HgS_2 \end{array}$$

In the presence of a large excess of HgS (such as that used in this method), one would expect all sodium sulphide to be present as the sodium mercury sulphide complex. Information as to the properties of this soluble complex is very scarce although Kolthoff, Griffith, and Moltzau (*4*) have published some work on the precipitation of mercuric sulphide from the complex by means of zinc sulphide. There is considerable room for argument as to the interchangeability of sodium sulphide and $Na_2HgS_2$ in the sodium sulphide hydrolysis equilibrium.

In an effort to detect any shift in the hydrolytic equilibrium, pH measurements were made on 0.1 *M* sodium sulphide solution before and after 24 hours of mixing with an excess of mercuric sulphide. The readings appear in Table VII.

**Table VII. pH of 0.1 Molar Sodium Sulphide (25°C.)**

| | *Initial pH* | *Final pH* |
|---|---|---|
| Run 1 | 12.90 | 12.83 |
| Run 2 | 12.91 | 12.86 |

A shift in the hydrolysis means a change in the sodium hydroxide concentration and, therefore, a change in the pH of the solution. Unfortunately, we are dealing with very high pH's and an extensive shift in the hydrolytic equilibrium would be required to shift the pH to an extent greater than the experimental error involved in a pH measurement. For 0.1 *M* sodium sulphide at pH 12.90, a shift of 16% in the hydrolysis equilibrium would only change the pH to 12.80. Table VII indicates that changes in pH of 0.07 and 0.05 were obtained experimentally. The magnitude of these changes is very close to the reproducibility of a pH measurement and, thus, too much significance cannot be attached to them. They may be interpreted as evidence that, if a shift in hydrolytic equilibrium exists, it does not exceed the order of

10%. The data in Tables V and IX, which present results as low as 1.3 and as high as 92.7% for the hydrolysis of sodium sulphide as determined by the Knox method, lend further support to the premise that the disruption of the hydrolytic equilibrium by complex formation is small. There is every reason to believe that, for sodium sulphide solutions more dilute than 0.15 *M*, the method will yield hydrolysis values higher than 92.7%.

***Degree of Completion of Settling of Excess Mercuric Sulphide***

It was desired to check the efficiency of the separation of excess mercuric sulphide from the supernatant liquid in the stainless steel tubes at elevated temperatures. A special piece of apparatus, shown in Fig. 3, was constructed to give a positive separation of the supernatant liquid from the excess mercuric sulphide at 100°C. The apparatus was so arranged that the supernatant liquid from a hydrolysis determination could be forced through an asbestos mat and sintered-glass disk in a filter stick by hydrogen pressure while the entire system was held in a boiling water bath. In this manner complete removal of the excess mercuric sulphide was insured. The filtered supernatant liquid was collected under a protective stream of nitrogen to minimize atmospheric oxidation and was analyzed for dissolved mercuric sulphide as previously described. The hydrolysis values obtained appear in Table VIII.

The average value of 65.0% hydrolysis obtained under these conditions compares well with that of 67.1% hydrolysis obtained for the same composition of solution at 110°C. (Table IX) by the regular high-temperature method. It is apparent that satisfactory removal of excess mercuric sulphide is obtained by the settling and decanting procedure used.

**Table VIII. Hydrolysis of Sodium Sulphide (100°C.) (Supernatant Liquid Filtered)**

| *Solution* | *Hydrolysis, %* |
|---|---|
| 0.2 *M* $Na_2S$, 0.5 *M* NaOH | |
| Run 1 | 64.9 |
| Run 2 | 65.1 |

## HYDROLYSIS OF SODIUM SULPHIDE IN SULPHIDE-HYDROXIDE MIXTURES

The hydrolysis of sodium sulphide at 25°C. and at elevated temperatures has been studied by the application of the mercuric sulphide solubility method of Knox. Table IX presents data on this hydrolysis at various sulphide and hydroxide concentrations and for several temperatures. The concentrations and temperatures were selected to cover the ranges normally encountered in sulphate pulping.

An examination of these data reveals that the sodium sulphide concentration and the temperature of the solution exert only minor influences on the degree of hydrolysis. The determining factor seems to be the amount of sodium hydroxide present, although the effect of this compound in suppressing the hydrolysis is also surprisingly small. From Table IX it appears that 1.5 *M* sodium hydroxide reduces the hydrolysis by little more than one half for the concentrations of sodium sulphide investigated. It was found that 8 or 9 molar sodium hydroxide was required to reduce the hydrolysis to less than 4% for dilute sodium sulphide solutions at 25°C.

Mitchell and Yorston (*10*), in experiments on the delignification of wood with sodium sulphide, found a direct proportionality between the rate of delignification and the sodium sulphide concentration. They considered this to be surprising, because of an expected change in the hydrolysis of sodium sulphide with concentration. The data of Table IX indicate that, at 25°C. at least, there is little change in the hydrolysis with change in sodium sulphide concentration over a concentration range of 0.15 *M* to 0.35 *M*.

**Table IX. Hydrolysis of Sodium Sulphide**

| M $Na_2S$ | *Molarity NaOH* 0, % | 0.25, % | 0.50, % | 0.75, % | 1.5, % |
|---|---|---|---|---|---|
| | | 25°C. | | | |
| 0.15 | 92.7 | 79.7 | 66.7 | 56.8 | 37.8 |
| 0.25 | 91.9 | 80.3 | .... | 58.7 | 41.0 |
| 0.35 | 83.8 | 71.9 | .... | 55.1 | 33.1 |
| | | 110°C. | | | |
| 0.20 | 89.3 | 79.3 | 67.1 | 51.9 | 43.1 |
| | | 165°C. | | | |
| 0.20 | 92.0 | 84.5 | 72.0 | 53.5 | 46.9 |

The small effect of temperature on the sodium sulphide hydrolysis, evidenced in Table IX, was contrary to expectation. It is well established that $K_w$, the ion product constant for water, shows a rapid increase with temperature. Since $K_h = K_w/K_a$, where $K_h$ is the hydrolysis constant for the salt and $K_a$ is the ionization constant of the acid of the salt, one might expect the hydrolysis to increase rapidly with $K_w$. Two possible reasons for the small change suggest themselves: (1) the increase of $K_w$ with temperature in a concentrated salt solution may be less than in the case of pure water and (2) the second ionization constant of hydrogen sulphide ($K_a$) may increase with temperature to such an extent as to balance the increase in $K_w$.

Unfortunately, data are not available in the literature on the variation of the second ionization constant of hydrogen sulphide with temperature. Jenkins (*5*) presents an equation for the ionization constant of acetic acid, which is satisfactory over a 50° temperature range. The equation is also applicable to formic, propionic, and butyric acids. Values of $1.751 \times 10^{-5}$ at 25°C. and $1.539 \times 10^{-5}$ at 60°C. were calculated for acetic acid.

Evidence for the idea that the second ionization of hydrogen sulphide increases with temperature is found when the heats of neutralization of certain weak acids are considered. The combination of hydrated hydrogen and hydroxyl ions to form water is accompanied by the evolution of 13.8 kilogram-calories per mole. In the neutralization of one mole of acetic acid by sodium hydroxide, 13.40 kilogram-calories are liberated. The difference between 13.8 and 13.40 is the heat consumed in the ionization of acetic acid in water. The neutralization of hydrogen cyanide ($K_a = 7 \times 10^{-10}$) by sodium hydroxide liberates only 2.90 kilogram-calories per mole. The lower the ionization con-

stant, the greater is the heat consumed in ionization of the acid, and one might expect that an acid with a high heat of ionization would exhibit an increase in ionization with increasing temperature. Thus, it seems quite possible that the second ionization of hydrogen sulphide ($K_2 = 3.63 \times 10^{-13}$) might be very sensitive to increased temperature.

## APPLICATION OF HYDROLYSIS DATA TO MILL COOKING DATA

It was felt that components of a sulphate black liquor other than sodium hydroxide would have little bearing on the sodium sulphide hydrolysis. To confirm this idea, the high-temperature adaptation of the Knox method was applied directly to some mill black liquors which had been analyzed for NaOH and total sulphide. The values obtained were of the same order of magnitude as the hydrolytic data on pure solutions of sodium hydroxide and sulphide, although the results obtained were somewhat erratic because of the coprecipitation of lignin when the black liquors were acidified to precipitate dissolved mercuric sulphide. Thorough washing of the precipitate with alcohol removed most of the lignin, but it was evident that the direct application of the mercuric sulphide method to black liquors yielded data of little value.

Since satisfactory data on the hydrolysis of the sodium sulphide in cooking liquors could not be obtained directly, the hydrolysis values for the various liquor samples were estimated by application of the data of Table IX on the hydrolysis of sodium sulphide in mixtures with sodium hydroxide to the total sulphide and sodium hydroxide contents of the liquor samples as determined by analysis. From these estimated hydrolysis values the total concentrations of sodium hydroxide, sodium sulphide, and sodium hydrosulphide existing in the solutions were calculated.

This procedure was applied to some analytical data on a Nekoosa-Edwards experimental cook as well as to those of a Mosinee mill-scale cook. The results obtained are tabulated in Tables X and XI and are

**Table X. Experimental Cooking Data (Nekoosa-Edwards)**

| *Time, min.* | *Free* M *NaOH* | *Total* M $Na_2S$ | *Estimated hydrolysis, %* | M *NaOH* | M $Na_2S$ | M *NaHS* |
|---|---|---|---|---|---|---|
| 0 | 1.30 | 0.37 | 38 | 1.44 | 0.23 | 0.14 |
| 60 | 0.48 | 0.28 | 64 | 0.66 | 0.10 | 0.18 |
| 120 | 0.27 | 0.27 | 78 | 0.48 | 0.06 | 0.21 |
| 180 | 0.17 | 0.26 | 85 | 0.39 | 0.04 | 0.22 |
| 240 | 0.11 | 0.26 | 88 | 0.34 | 0.03 | 0.23 |

plotted in Figs. 4 and 5. The term "*M* NaOH (free)" used in these tables and figures does not include sodium hydroxide formed by hydrolysis of the sodium sulphide.

The original analytical data on the Mosinee cook are indicated by the points in Fig. 5, whereas those for the Nekoosa-Edwards cook were taken from Borlew and Pascoe (*6*).

## SODIUM SULPHIDE HYDROLYSIS IN SULPHATE COOKING

Many assumptions as to the hydrolysis of sodium sulphide in a sulphate cooking liquor appear in the literature. Bray, Martin, and Schwartz (*7*) and Hanson (*8*), on one hand, assumed complete or practically complete hydrolysis of the sodium sulphide to sodium hydroxide and sodium hydrosulphide. On the other hand, Borlew and Pascoe (*6*) assumed zero hydrolysis, stating that sodium hydrosulphide does not exist in a sulphate cooking liquor and, furthermore, that it cannot exist at a pH higher than 8.3. The data of Table IX indicate an appreciable hydrolysis, and substantiate Brauns' (*9*) idea of a partial hydrolysis of sodium sulphide to hydrosulphide, from which compound a mercapto group is formed in sulphate lignin. It should be mentioned again that these data were taken on mixtures of pure solutions of sodium sulphide and sodium hydroxide and not upon actual cooking liquors. It is felt, however, that the other components in a cooking liquor, or even in a black liquor, would have little effect upon the sodium sulphide hydrolysis.

**Table XI. Mill-Scale Cooking Data (Mosinee)**

| *Time, min.* | *Free* M *NaOH* | *Total* M $Na_2S$ | *Estimated hydrolysis, %* | M *NaOH* | M $Na_2S$ | M *NaHS* |
|---|---|---|---|---|---|---|
| 0 | 1.77 | 0.26 | 42 | 1.88 | 0.15 | 0.110 |
| 35 | 0.98 | 0.13 | 57 | 1.05 | 0.06 | 0.074 |
| 70 | 0.80 | 0.11 | 65 | 0.87 | 0.04 | 0.072 |
| 105 | 0.67 | 0.10 | 73 | 0.74 | 0.03 | 0.073 |
| 135 | 0.58 | 0.10 | 76 | 0.66 | 0.02 | 0.076 |

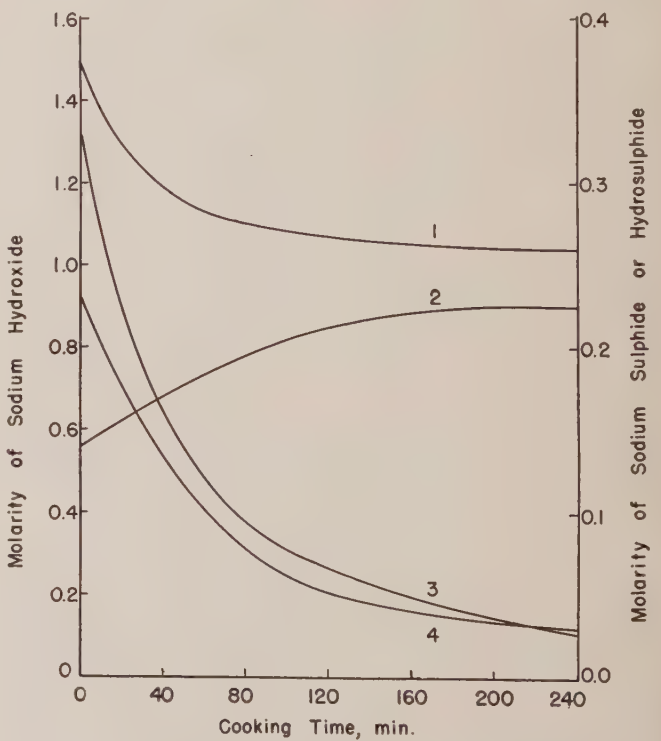



***Fig. 4. Experimental cooking data (Nekoosa-Edwards)***

(1) *M* $Na_2S$ (total) (by analysis), (2) *M* NaHS (by calculation), (3) *M* NaOH (free) (by analysis), and (4) *M* $Na_2S$ as $Na_2S$ (by calculation).

Table X and Fig. 4 present analytical data from an experimental cook to which has been added an estimation of the sodium sulphide hydrolysis, based upon the data of Table IX. Some interesting observations in connection with various ideas that have been advanced

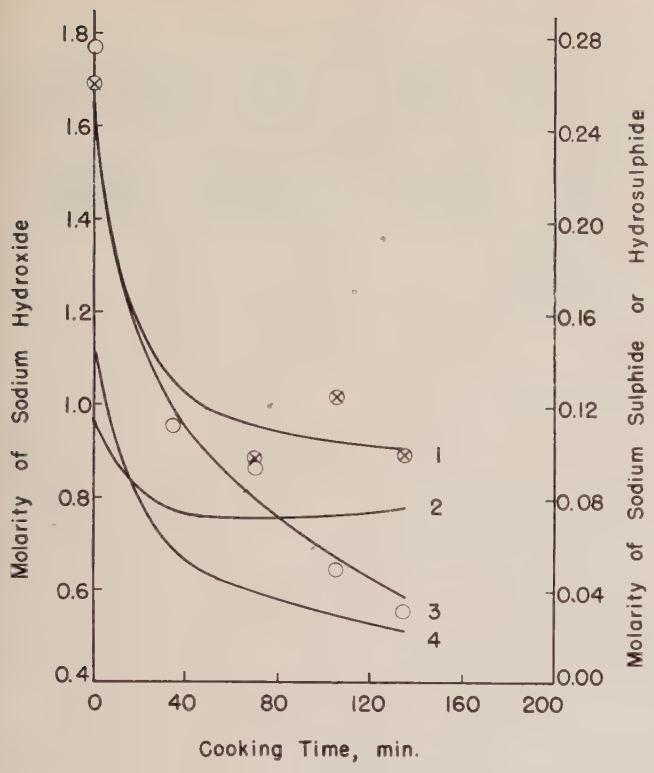



***Fig. 5. Mill-scale cooking data (Mosinee)***

(1) *M* $Na_2S$ (total) (by analysis), (2) *M* NaHS (by calculation), (3) *M* NaOH (free) (by analysis), and (4) *M* $Na_2S$ as $Na_2S$ (by calculation).

on sulphate pulping can be made by considering the hydrolysis at several points during a typical cook.

Let us consider the cook described in Table X as a typical sulphate cook, in which the temperature is raised to 165°C. in 1.5 hours and held there for 2.5 hours before the digester is relieved and blown.

When the cook is started, the sodium sulphide is only 38% hydrolyzed; the total concentrations in solution are 1.44 *M* sodium hydroxide, 0.23 *M* sodium sulphide, and 0.14 *M* sodium hydrosulphide. During the first hour air is removed from the digester, the liquor penetrates the chips and the volatile oils distill off with the steam. The more soluble carbohydrates are attacked to an extent greater than the lignin during the very early part of the cook. The entrance of sulphur into the lignin, with the resulting increase of lignin solubility in alkali, may take a little time to reach appreciable proportions. The sulphur apparently enters the lignin by reaction with the sodium hydrosulphide and forms a mercapto group.

$$C{=}O + HSNa \longrightarrow C\begin{matrix}OH\\SNa\end{matrix}$$

It is the presence of this mercapto group that is responsible for the increase in alkali solubility.

At the end of the first hour the temperature is in the neighborhood of 140°C., and the sodium sulphide is 64% hydrolyzed. The concentration of sodium hydrosulphide in solution has actually increased to 0.18 *M*, whereas the sodium hydroxide is 0.66 *M* and the sodium sulphide is 0.10 *M*. It is in this region that the rate of delignification and the ratio of lignin to carbohydrates dissolved are approaching their maximums.

At the end of the second hour, maximum temperature has been reached and the sodium sulphide is 78% hydrolyzed. The concentrations in solution are now 0.48 *M* for sodium hydroxide, 0.06 *M* for sodium sulphide, and 0.21 *M* for sodium hydrosulphide. The evolution of noncondensable gases has practically ceased and the more readily accessible lignin has been removed.

During the last 2 hours of the cook, there is a slow lignin removal and some degradation of the cellulose. The cooking liquor is rich in sodium hydrosulphide but the alkali concentration has become rather low. Much of the lignin now being removed is from the middle lamella. This lignin may have been rendered less soluble by condensation reactions earlier in the cook.

Theoretical support for the recognized benefits of preimpregnation with black liquor, as well as for sulphate injection pulping, is found in the hydrolysis and cooking data. From Table X it is evident that the black liquor has a high concentration of sodium hydrosulphide. Sulphur may enter the lignin in appreciable amounts during the preimpregnation period. The thiolignin formed will then dissolve rapidly as soon as the white liquor, rich in sodium hydroxide, comes in contact with the wood.

In injection cooking, a more dilute liquor at the beginning of the cook should eliminate or materially reduce the period during which carbohydrates are dissolved at a more rapid rate than the lignin. It is suggested that maximum benefits might be obtained by starting the cook with a high-sulphidity white liquor in the digester, and injecting a liquor of lower sulphidity and higher sodium hydroxide content as the cook progresses. In this way, a high concentration of sodium hydrosulphide would be present to react with the lignin during the early stages, and a sufficient amount of sodium hydroxide would be present toward the end of the cook to dissolve the more resistant lignin.

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# The Characteristics of Unbleached Kraft Pulps from Western Hemlock, Douglas Fir, Western Red Cedar, Loblolly Pine, and Black Spruce

## *I. The Chemical Constituents of the Unbleached Kraft Pulps*

HARRY F. LEWIS, LOUIS E. WISE, and B. L. BROWNING

**In this first paper of the series devoted to a better understanding of possible causes for the differences between the physical properties of the kraft pulps from Douglas fir and loblolly pine, on the one hand, and from western hemlock, black spruce, and western red cedar, on the other, a study of their chemical components is described. The results fail to show significant chemical variations which might account adequately *per se* for the differences in physical properties. Some variations in composition have been shown, as might be expected. The spruce pulp, for example, shows the lowest corrected alpha-cellulose content—on a corrected basis, western hemlock and Douglas fir pulps have almost identical alpha-cellulose contents. The lignin contents are approximately the same, confirming that about the same degree of delignification was achieved in the cooking of each wood. Spruce and fir pulps had similar mannan contents, with hemlock intermediate. In the case of the fir pulp, this mannan was found almost entirely in the alpha-cellulose fraction. Fir and hemlock pulps had much the same uronic anhydride contents. These same pulps had the lowest pentosan (xylan) contents.**

In a study of the kraft pulping of western hemlock, Douglas fir, western red cedar, loblolly pine, and black spruce (*1*), various differences were observed between the pulps from Douglas fir and loblolly pine, on the one hand, and those from western red cedar, western hemlock, and black spruce, on the other. The present work was undertaken to explain, if possible, the characteristic differences in these properties.

Among the approaches to this problem were (1) a chemical examination of the pulps, (2) a morphological study of the fibrous components, (3) a study of the specific surface area of the pulps and the change in this area on beating, (4) a study of the distribution of the cellulose in the pulps by degree of polymerization, and (5) an investigation of the x ray orientation of the fibers.

The present paper contains a discussion of the first approach—i.e., the chemical examination of the pulps. Additional papers in this series will discuss the other approaches.

### CHEMICAL ANALYSES

The pulps were received in an air-dry shredded condition. Samples were prepared by standard methods for the determination of extractives, lignin, and ash. The carbohydrate components were determined by chloriting 5-gram samples; these holocellulose residues were then converted to alpha-cellulose and hemicelluloses A and B in the usual manner (cf. *1*).

The principal components of the kraft pulp are summarized in Table I.

**Table I. Composition of the Kraft Pulps from the Five Woods**

| *Pulp* | *Alpha-cellulose, %* | *Hemi-cellulose A, %* | *Hemi-cellulose B, %* | *Lignin, %* | *Ash, %* | *Extractives, %* |
|---|---|---|---|---|---|---|
| Fir | 87.6 | 2.8 | 3.2 | 5.8 | 0.7 | 0.6 |
| Hemlock | 86.6 | 3.4 | 3.7 | 5.3 | 1.0 | 0.6 |
| Cedar | 85.4 | 3.7 | 3.2 | 6.2 | 0.6 | 0.8 |
| Pine | 85.1 | 4.8 | 3.0 | 5.4 | 0.5 | 0.5 |
| Spruce | 82.5 | 5.1 | 3.7 | 5.8 | 0.6 | 0.7 |

The chemical analyses of the pulps failed to show any significant differences which might account adequately for the physical properties of the fir and pine pulps. Very similar values for lignin indicate that the cooking conditions followed resulted in much the same degree of delignification, a fact already known from the permanganate number of the pulp. The high alpha-cellulose content of the fir emphasizes a point raised by Brookbank (*2*) that Douglas fir might be a good wood for the manufacture of chemical pulps because of the high alpha-cellulose content of the Douglas fir sulphite pulp.

The summative analyses given in Table II include the values for alpha-cellulose (corrected for mannan, xylan, and uronic anhydride), lignin, ash, extractives, mannan, xylan, and uronic anhydride.

**Table II. Summative Analyses of the Five Pulps**

| | *Black spruce* | *Loblolly pine* | *Western red cedar* | *Douglas fir* | *Western hemlock* |
|---|---|---|---|---|---|
| Alpha-cellulose (corrected) | 75.2 | 78.6 | 78.7 | 79.8 | 79.9 |
| Lignin | 5.8 | 5.4 | 6.2 | 5.8 | 5.3 |
| Ash | 0.6 | 0.5 | 0.6 | 0.7 | 1.0 |
| Extractives | 0.7 | 0.5 | 0.8 | 0.6 | 0.6 |
| Mannan | 5.0 | 2.6 | 2.8 | 5.2 | 3.8 |
| Xylan | 7.9 | 8.3 | 7.3 | 4.9 | 5.6 |
| Uronic anhydride | 1.8 | 1.6 | 1.5 | 2.3 | 2.1 |
| Total | 97.0 | 97.5 | 97.9 | 99.3 | 98.3 |

The data show that Douglas fir and western hemlock pulps have almost identical alpha-cellulose con-

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tents, and consequently the yields of cellulose derivatives from the alpha-cellulose would be much the same for fir and hemlock and only slightly less for pine and cedar. The corrected alpha-cellulose content for spruce, however, is significantly lower. As in the case of the chemical analyses, the summative analysis fails to exhibit any significant differences which might account for the physical properties of the fir and pine pulps. One observation is that fir pulp has a high-mannan content and a low-xylan content, whereas the pine pulp has a high-xylan content and a low-mannan content. Low-xylan contents are noted for both fir and hemlock, in contrast to spruce, pine, and cedar, whereas low-mannan contents are noted for pine, cedar, and hemlock. The uronic anhydride contents are likewise low for spruce, pine, and cedar in comparison with fir and hemlock. Chemically, the hemlock and fir pulps appear to be more closely related than are the pine and fir pulps.

| | *Weight (oven-dry) sulphate pulp taken, gram* | *Weight (oven-dry) holocellulose recovered, gram* | *Holocellulose based on original oven-dry pulp, %* | *Holocellulose[a] based on oven-dry pulp (from small 5-gram samples), %* | *Holocellulose calculated from sum of hemicelluloses A and B and alpha-cellulose, %* |
|---|---|---|---|---|---|
| Douglas fir | 47.11 | 44.60 | 93.9 | 94.55 | 93.5 |
| Western hemlock | 47.8 | 44.51 | 93.1 | 94.3 | 93.6 |

[a] Probably still retains small amounts of lignin.

Other analytical data are included in Table III, which gives the mannan, xylan, and uronic anhydride components of the holocellulose and the alpha-celluloses; these data are also recalculated on the basis of the *original* oven-dried unbleached pulps (assuming 94% yields of holocellulose).

It is interesting to note that the mannan appears to be almost entirely associated with the alpha-cellu-

**Table III**

Components of Holocellulose from Sulphate Pulps

| *Pulp* | *Mannan* | | *Xylan* | | *Uronic anhydride* | |
|---|---|---|---|---|---|---|
| | *Based on holocellulose, %* | *Based on unbleached pulp, %* | *Based on holocellulose, %* | *Based on unbleached pulp, %* | *Based on holocellulose, %* | *Based on unbleached pulp,[a] %* |
| Douglas fir | 5.4 | 5.08 | 5.25 | 4.94 | 1.95 | 2.26 |
| Western hemlock | 4.1 | 3.75 | 5.98 | 5.63 | 1.95 | 2.08 |

Components of Alpha-cellulose (From Holocellulose from Pulps)

| | *Mannan* | | *Xylan* | | *Uronic Anhydride* | |
|---|---|---|---|---|---|---|
| | *Based on alpha-cellulose, %* | *Based on unbleached pulp, %* | *Based on alpha-cellulose, %* | *Based on unbleached pulp, %* | *Based on alpha-cellulose, %* | *Based on unbleached pulp, %* |
| Douglas fir | 5.7 | 4.99 | 1.60 | 1.44 | 1.4 | 1.25 |
| Western hemlock | 4.6 | 3.98 | 1.65 | 1.39 | 1.5 | 1.29 |

Components of the Total Isolated Hemicellulose (Calculated) Based on Original Unbleached Pulps

| | *Mannan* | *Xylan, %* | *Uronic anhydride, %* |
|---|---|---|---|
| Douglas fir | None | 3.50 | 1.01 |
| Western hemlock | None | 4.24 | 0.79 |

[a] These were separate determinations made on the original unbleached pulp (*not* calculated from holocellulose).

In order to determine whether the pectic acid content of the pulps might not cause some of the difference in the beating characteristics of the pulps, particularly in the development of bursting strength, E. Anderson made extensive analyses of the pulps for pectic acid. The maximum content of pectic acid is not more than a few thousandths of a per cent; hence, this component can be eliminated as being responsible for the properties of these kraft pulps.

## HOLOCELLULOSE FRACTIONS

A more extensive study was made of the holocellulose fractions from the western hemlock and Douglas fir pulps. In this study, large-scale holocellulose preparations were made quantitatively under the following conditions: About 50 grams of air-dried, unbleached, shredded sulphate pulp samples were heated for 35 minutes with 1600 ml. of water, 15 grams of sodium chlorite, and 5 ml. of acetic acid at 60 to 70° with constant stirring. At the end of this period, the mixtures were cooled in ice water, washed with ice water and acetone, and air dried. Moisture determinations were then made and the oven-dry weight calculated.

The results may be tabulated as follows:

lose of the pulp. The results confirm the results for the original Douglas fir and western hemlock woods from which these pulps were prepared. From previous findings by Anderson, it is probable that small amounts of mannan are in the hemicellulose. Even if this is the case, it is doubtful whether the greater part of the mannan behaves as a "hemicellulose" in its effects on pulp quality and, thus, an "excess of mannan" in the case of Douglas fir may be without significance other than in increasing the apparent "alpha-cellulose" content of the pulp.

The significance of mannan in pulp needs further study. In 1937 Brauns and Lewis (*3*) described the nature of the insoluble residue remaining after a bleached sulphite pulp had been treated with cuprammonium solution; the insoluble material had the outline of the somewhat swollen fiber when photographed in a dark field. The product seemed to consist of a copper carbohydrate complex containing 30% copper. The remaining material was carbohydrate in nature. When the copper was removed with ammonium hydroxide, followed by a weak acid treatment, the carbohydrate portion was a white gelatinous mass yielding a white starchlike product on drying, which had a carbon content only slightly less than that of cellu-

lose—44.2% instead of 44.44%. In the earlier analysis, qualitative tests for pentosans, mannans, and galactans in the acid-hydrolyzed material were negative. Recently, a chromatographic separation of this acid hydrolyzate indicated a high percentage of mannose. This was substantiated by the Analytical Laboratory of the Institute which found the simple sugars to be approximately three quarters mannose. It was previously reported that the product would not form a soluble acetate when acetylated with pyridine and acetic anhydride. It is possible that the mannan in the outer wall of the fiber may be a partial cause of the haze of acetate solutions prepared from wood pulp. The relation of mannan to the papermaking properties of pulp is not known, but further study seems warranted.

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# The Characteristics of Unbleached Kraft Pulps from Western Hemlock, Douglas Fir, Western Red Cedar, Loblolly Pine, and Black Spruce

## *II. The Morphological Characteristics of the Pulp Fibers*


JOHN H. GRAFF and IRVING H. ISENBERG



**Data are given for the fiber length and width of unbleached kraft pulps for western hemlock, Douglas fir, western red cedar, loblolly pine, and black spruce.**


Morphological studies have been made of wood and pulp fibers from the five woods listed in the abstract, with particular emphasis being placed upon a variety of measurements with western hemlock and Douglas fir.

The following information has been selected from a large number of studies made at the Institute. Unbroken fibers of western hemlock range in length from 1.3 to 6.3 mm., with an average of 4.2. Fibers of Douglas fir range from 1.7 to 7.0 mm., with an average of 3.9 mm. The range in width is 30 to 40 mu for western hemlock (widest 45 mu) and 35 to 45 mu for Douglas fir (widest 55 mu). Thus, the two woods have much the same average fiber length. The fir fibers average 15 to 25% wider than the hemlock fibers.

The obvious anatomical differences between the two species are the abrupt transition from springwood to summerwood, the presence of normal resin canals, the occurrence of spiral thickening on the inside of the tracheid walls, and the greater tangential diameter of the tracheids, which are all characteristic of Douglas fir.

The distribution of fibers by length and thickness is shown in Fig. 1. The differences in the distribution of fiber lengths seem to be highly significant. The greater thickness of the Douglas fir fibers would indicate a smaller fiber surface area for fiber-to-fiber contact in the sheet. The spiral thickening might affect the flexibility and stretch of the fiber. The presence of a large amount of thick-walled summerwood fibers would also result in fewer fibers for a given weight of pulp stock. In the lower half of the figure, the relation between cell wall thickness and frequency is shown. The solid line for the Douglas fir

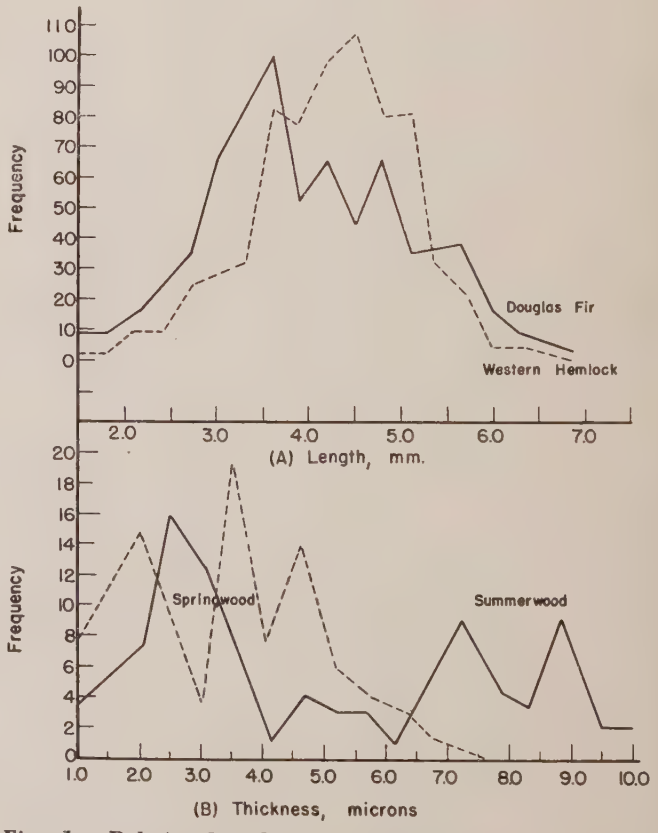


*Fig. 1. Relationship between frequency and (a) fiber length and (b) cell wall thickness*


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Table I. Summary of Fiber Dimensions of Unbleached Kraft Pulps

| | *Douglas fir* | *Loblolly pine* | *Western hemlock* | *Spruce*[a] | *Western red cedar* |
|---|---|---|---|---|---|
| Fiber length, mm. | | | | | |
| Arithmetical average | 2.62 | 2.54 | 2.21 | 2.14 | 2.05 |
| Weighted average | 3.38 | 3.17 | 2.8 | 2.86 | 2.76 |
| Fiber width, mm. | 0.044 | 0.043 | 0.041 | 0.037 | 0.040 |
| Area, sq. mm. | 0.112 | 0.113 | 0.100 | 0.084 | 0.089 |
| Fibers per gram, millions | 3.5 | 3.8 | 4.5 | 4.3 | 4.5 |

[a] Bleached kraft.

shows the presence of large numbers of fibers having a cell wall thickness of between 2 and 3 mu and also a fairly large number of fibers of cell wall thickness between 7 and 9 mu; the thin fibers are the springwood, the thicker fibers the summerwood. Western hemlock, on the other hand, fails to show this difference.

The fiber dimensions of unbleached kraft pulps prepared from Douglas fir, loblolly pine, western hemlock, and western red cedar, together with data on the fiber dimensions of bleached spruce kraft fibers, are summarized in Table I. The results show that the average fiber length for Douglas fir and loblolly pine exceeds that for the other three pulps, and the same might be said for the fiber widths, although the differences between the various pulps is not too great. The fiber areas show that the Douglas fir and loblolly pine fibers have the greatest areas; western hemlock is next, and spruce and western red cedar have the lowest values. The number of fibers per gram of pulp will be somewhat related to the fiber areas but in a reverse order. Western hemlock and western red cedar have approximately 4.5 million per gram, spruce is slightly less with 4.3 million, whereas loblolly pine is lower with 3.8 million, and Douglas fir is the lowest with only 3.5 million.

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# The Characteristics of Unbleached Kraft Pulps from Western Hemlock, Douglas Fir, Western Red Cedar, Loblolly Pine, and Black Spruce

## *III. The Comparative Pulping of the Woods*

W. F. HOLZER and K. G. BOOTH

**Five wood species were pulped by the Central Research and Technical Department of Crown Zellerbach Corp. in the study of pulpwoods carried out in conjunction with The Institute of Paper Chemistry. Both kraft and sulphite pulps were prepared from Douglas fir, western hemlock, western red cedar, eastern black spruce, and loblolly pine. The selected logs were handbarked and chipped in commercial chippers, then air dried. Kraft cooks were made in an experimental rotary digester to a TAPPI permanganate No. of 30. Hemlock was the slowest pulping species, while eastern spruce pulped quite easily. The other three species were intermediate, and quite close in ease of pulping. Hemlock, spruce, and pine yields were quite similar, whereas fir and cedar gave much lower yields. Eastern spruce had shorter fibers than the other four species which were quite similar in fiber classifications. Cedar pulp gave the highest bursting strength, followed closely by spruce and hemlock, with Douglas fir and loblolly pine giving much lower values. The order of decreasing tear resistance was substantially the reverse of the bursting strengths. The sulphite pulps were prepared in an experimental digester fitted with forced circulation and indirect heating. Calcium base acid was used for cedar, hemlock, and spruce but sodium-base acid was necessary for the other species to prevent burning. Higher temperatures and longer cooking cycles were needed for cedar and Douglas fir to obtain pulps with TAPPI permanganate Nos. of 15. Yields of pulp were substantially the same. Fiber classifications were closely similar for all pulps except the shorter fibered spruce. The orders for decreasing bursting and tear strengths were nearly the same as in the kraft cooks. Hemlock and eastern spruce are the only two of these five species normally used in sulphite pulping, and the other three species produced quite dark pulps in these cooks.**

The physical properties of pulps are, in the ultimate, dependent on the wood species used. The maximum value which can be obtained for any given physical property is limited by the properties of the original wood fiber. Pulping of different wood species under rigorously standardized laboratory conditions will show, in a comparative though not absolute manner, the pulp properties which are inherent in those species. This report gives an account of the comparative pulping of five pulpwood species of which three were western woods: western hemlock (*Tsuga heterophylla*), Douglas fir (*Pseudotsuga taxifolia*), and western red cedar (*Thuja plicata*), one an eastern

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wood: eastern black spruce (*Picea mariana*), and the fifth a southern wood: loblolly pine (*Pinus taeda*).

This work formed part of an intensive investigation of the five species carried out by The Institute of Paper Chemistry at Appleton, Wis., in cooperation with the Central Research and Technical Department of Crown Zellerbach Corp. The pulping of the woods and the determination of physical properties of the pulps was done by the latter organization. Samples of all pulps and also of the waste liquors were sent to The Institute of Paper Chemistry for microscopic and chemical investigation.

The wood samples used were selected as being typical of the species, and were free of decay. They had been floated in water for only a short space of time. Their origins were as follows:

| | | |
|---|---|---|
| Western hemlock | Siltcoos area near mouth of Umpqua River Elevation 150 ft. | Crown Zellerbach Corp. |
| Douglas fir | Willamette Valley | Crown Zellerbach Corp. |
| Western red cedar | North fork of Lewis River, north of Kalama | Crown Zellerbach Corp. |
| Loblolly pine | Eastern North Carolina | North Carolina Pulp Co. |
| Eastern black spruce | Probably from Canada, area of the Soo | Interlake Mill of Consolidated Water Power and Paper Co. |

The selected logs or blocks were barked by hand and then chipped in the commercial chippers of the Crown Zellerbach mill at Camas, Wash. Pulping of the chips in the experimental kraft and sulphite digesters was carried out under carefully standardized conditions with the object of obtaining kraft pulps with a TAPPI permanganate number of 30, and sulphite pulps with a permanganate number of 15.

## KRAFT PULPING

The kraft cooks were made in a rotary digester of 2.4 cubic foot capacity, heated by a steam jacket. Because of the large ratio of digester shell to digester contents, it was impossible to use direct steam as in the commercial process since the amount of condensate would have been excessive. Compensation for the lack of liquor volume without condensate was made by adding extra water to the digester at the beginning of the cook. The chemical usage was 14% of active alkali, calculated as sodium oxide and based on the moisture-free wood weight. The sulphidity averaged 26%. In all cooks the time of rise to maximum temperature was 45 minutes, and the maximum temperature chosen was 355°F. To obtain pulps of the desired hardness mentioned above, the time at maximum temperature was varied for each species of wood. The results are given in Table I, and inspection shows that western hemlock required the longest time at temperature for pulping. Cedar, loblolly pine, and Douglas fir were similar in ease of pulping, although had all three pulps been exactly equal in bleachability there would have been a slightly greater range in the times at temperature. Eastern spruce was easily the most rapid pulping species of the five.

Hemlock, spruce, and pine yields were very similar, with fir and cedar ranking much lower. One reason for the low yield from cedar lies in the high percentage of water extractives in this wood, and moreover it has a high lignin content. The screenings yields, except for hemlock, were abnormally high, though this can be accounted for because of the use of air-dried chips, a procedure which usually leads to high screenings. The chips used in this investigation were air dried to decrease deterioration on storage.

**Table I. Kraft Pulping of the Five Wood Species**

| *Species* | *Western hemlock* | *Douglas fir* | *Cedar* | *Eastern spruce* | *Loblolly pine* |
|---|---|---|---|---|---|
| Time at maximum temperature, min. | 54 | 46 | 48 | 39 | 47 |
| Total time, min. | 99 | 91 | 93 | 84 | 92 |
| Screened yield, % | 44.8 | 42.7 | 41.8 | 45.2 | 44.5 |
| Screenings, % | 4.7 | 10.0 | 8.2 | 8.2 | 6.5 |
| TAPPI permanganate No. | 29.6 | 30.1 | 31.6 | 31.5 | 28.2 |

All five pulps were tested for fiber classification, using the Bauer-McNett Classifier. Table II shows the results, which appeared normal for the various species, except that hemlock pulps are usually retained to the extent of 81% on the 20-mesh screen, rather than 79%. Note that the eastern spruce had shorter fibers, on the average, than the other four species.

**Table II. Fiber Classifications of the Kraft Pulps**

| *Species* | *Western hemlock* | *Douglas fir* | *Cedar* | *Eastern spruce* | *Loblolly pine* |
|---|---|---|---|---|---|
| On 20 mesh | 79.0 | 81.6 | 80.9 | 75.2 | 82.5 |
| On 35 mesh | 12.5 | 9.9 | 11.8 | 14.4 | 11.0 |
| On 65 mesh | 3.5 | 3.2 | 4.5 | 4.4 | 3.4 |
| On 150 mesh | 1.5 | 1.1 | 1.2 | 1.3 | 0.9 |
| Through 150 mesh | 3.5 | 4.2 | 1.6 | 4.7 | 2.2 |

The pulps were tested according to TAPPI procedures, using a Valley beater. The burst strengths and tear resistances are given in Table III and the respective beater curves, together with freeness curves, in Figs. 1 and 2. The cedar pulp had a very high burst strength, then came eastern spruce and hemlock which were not much lower. Douglas fir and loblolly pine developed considerably less bursting strength. As is generally the case, the tear resistance decreased in substantially the opposite direction with Douglas fir having the highest value, then loblolly pine, western hemlock, cedar, and eastern spruce in that order. Beating times to 400 ml. Canadian Standard freeness decreased in almost the same order as the burst values.

## SULPHITE PULPING

Of the five woods, only two, western hemlock and eastern spruce, are generally used in sulphite pulping, but all five were tested for the purpose of comparison. The pulping was carried out in the experimental sulphite digester which is fitted with forced circulation and indirect heating and has automatic pressure and temperature controls. The liquor to moisture-free wood ratio varied from 6:1 to 6.5:1, depending on wood density and moisture in the chips. Calcium-base acid was used for cedar, hemlock, and spruce, but for Douglas fir and loblolly pine sodium base acid was necessary since, in the latter stages of the cooks with

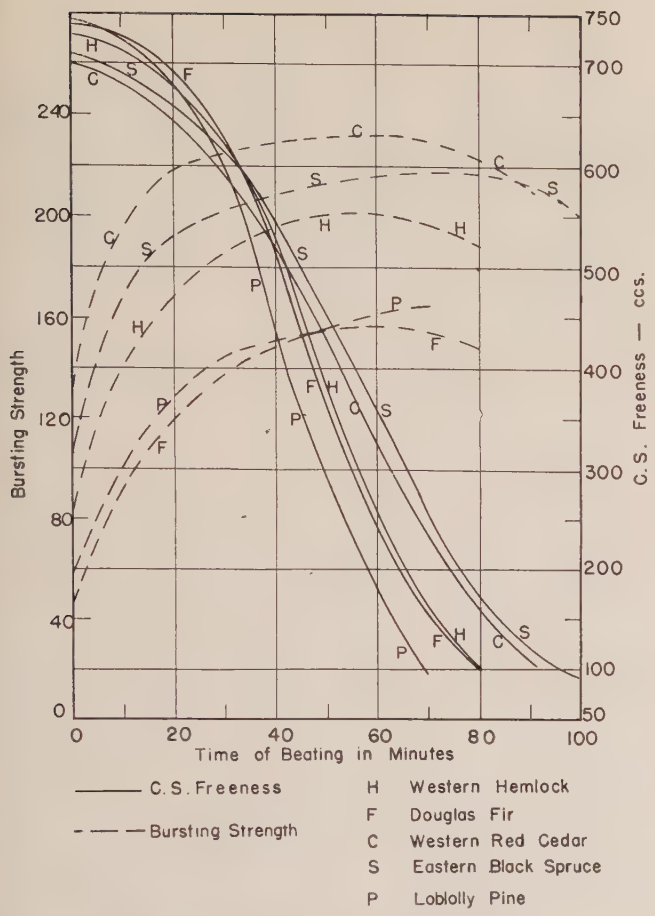


***Fig. 1. Bursting strengths of kraft pulps***

calcium base acid, the calcium is precipitated out and the cooks become burned. However, many examples are available in the literature to show that the substitution of sodium for calcium under comparable conditions produces very little change in pulp qualities.

**Table III. Physical Testing of the Kraft Pulps**

| *Species* | *Western hemlock* | *Douglas fir* | *Cedar* | *Eastern spruce* | *Loblolly pine* |
|---|---|---|---|---|---|
| Burst, % | | | | | |
| at 600 ml. freeness | 189 | 142 | 223 | 204 | 143 |
| at 400 ml. freeness | 200 | 154 | 231 | 214 | 152 |
| Tear resistance | | | | | |
| at 600 ml. freeness | 1.80 | 2.95 | 1.57 | 1.43 | 2.48 |
| at 400 ml. freeness | 1.74 | 2.65 | 1.53 | 1.36 | 2.20 |
| Time to: | | | | | |
| 600 ml. freeness | 33 | 33 | 28 | 33 | 30 |
| 400 ml. freeness | 48 | 47 | 53 | 55 | 42 |

Separate acid was made up for each cook, no relief from a previous cook being used in order to prevent the carry-over of volatiles or entrained liquor from one cook to the next. The combined sulphur dioxide was held to 1.20 ± 0.02% in all cases, and the total sulphur dioxide to 7.0 ± 0.2% for hemlock, Douglas fir, and cedar. Total sulphur dioxide in the pine cooks averaged 6.70%, and in the eastern spruce cooks 7.70%. As in the kraft cooks previously mentioned, a standard time of rise to temperature, in this case 4.5 hours, was used for all species. A maximum temperature of 275°F. was chosen for hemlock, spruce, and loblolly pine. However, the other two woods pulp very slowly at this temperature, and maximum temperatures of 280° for cedar, and 285° for Douglas fir were chosen. The cooking data are shown in Table IV. It had been anticipated that loblolly pine would cook about the same as Douglas fir, but with precipitation of the base eliminated by the use of sodium, an extremely soft pulp was produced on the Douglas fir cycle. Repeating the cook on the cycle used for spruce gave a pulp of proper hardness.

The total cooking cycles for spruce and pine were 8 hours, hemlock required 8.5 hours, and Douglas fir and cedar 9.5 and 9.75 hours, respectively, despite the higher temperatures used for the two latter species. The maximum pressure was held to 95 p.s.i., this pressure being reached 3 to 3.5 hours after the beginning of the cook. The blow down to a blowing pressure of 30 p.s.i. was started 1.25 hours before the completion of the cook.

**Table IV. Sulphite Pulping of the Five Wood Species**

| *Species* | *Western hemlock* | *Douglas fir* | *Cedar* | *Eastern spruce* | *Loblolly pine* |
|---|---|---|---|---|---|
| Maximum temperature, °F. | 275 | 285 | 280 | 275 | 275 |
| Total time, hours | 8.5 | 9.5 | 9.75 | 8.0 | 8.0 |
| Screened yield, % | 45.0 | 45.2 | 45.5 | 45.0 | 47.1 |
| Screenings, % | 3.3 | 1.8 | 2.8 | 2.5 | 6.5 |
| TAPPI permanganate No. | 15.1 | 12.5 | 16.3 | 14.1 | 15.6 |

The screened pulp yields were all close to 45% based on moisture-free wood, except for loblolly pine which gave a yield of 47%. No comparative data

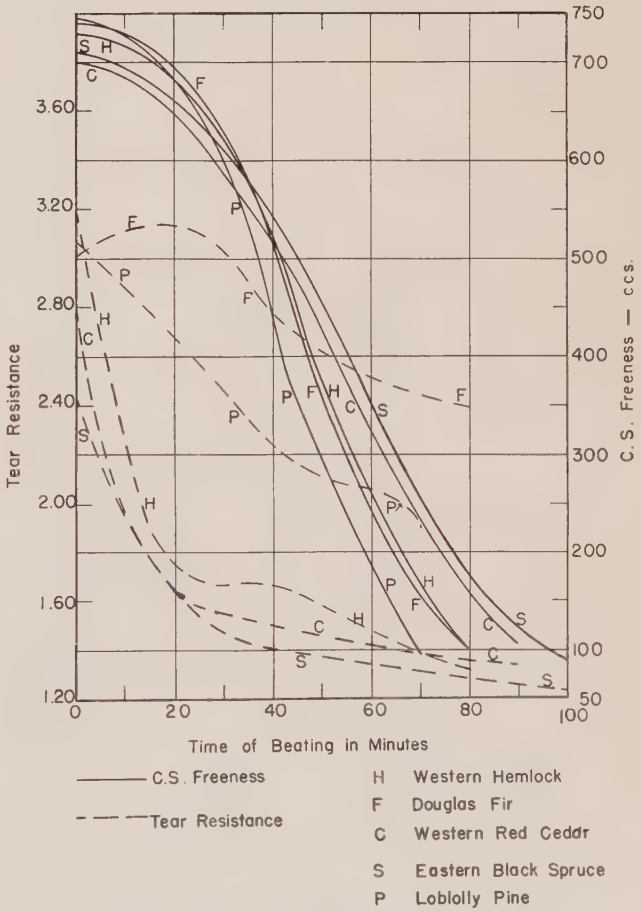


***Fig. 2. Tear resistances of kraft pulps***

are available for this latter species. The yield from this wood in kraft pulping was good, but no higher than from other woods. The cedar yield is rather higher than would be expected for the reasons previously mentioned, while hemlock generally averages 46% yield. As in the kraft cooks, the screenings appeared to be on the high side, but again the fact that the chips had been air-dried seems a reasonable explanation for the difference.

Fiber classifications on these pulps are shown in Table V. As in the kraft pulps, the eastern spruce yielded a smaller coarse fraction in keeping with the generally shorter fiber length of this species. The coarse fraction from the other pulps lay in a small range, from 70.0 to 71.7%.

**Table V. Fiber Classifications of the Sulphite Pulps**

| *Species* | *Western hemlock* | *Douglas fir* | *Cedar* | *Eastern spruce* | *Loblolly pine* |
|---|---|---|---|---|---|
| On 20 mesh | 71.7 | 70.1 | 70.0 | 68.5 | 71.5 |
| On 35 mesh | 16.1 | 13.1 | 15.0 | 16.7 | 14.4 |
| On 65 mesh | 5.3 | 6.8 | 7.3 | 5.5 | 5.9 |
| On 150 mesh | 2.7 | 2.5 | 3.0 | 2.1 | 2.1 |
| Through 150 mesh | 4.2 | 7.5 | 4.7 | 7.2 | 6.1 |

Physical strength tests on the different pulps showed almost identical trends to those obtained with the kraft pulps, as shown in Table VI and Figs. 3 and 4. Cedar again had the highest burst strength, with hemlock and eastern spruce having somewhat lower and equal values. Douglas fir and loblolly pine had

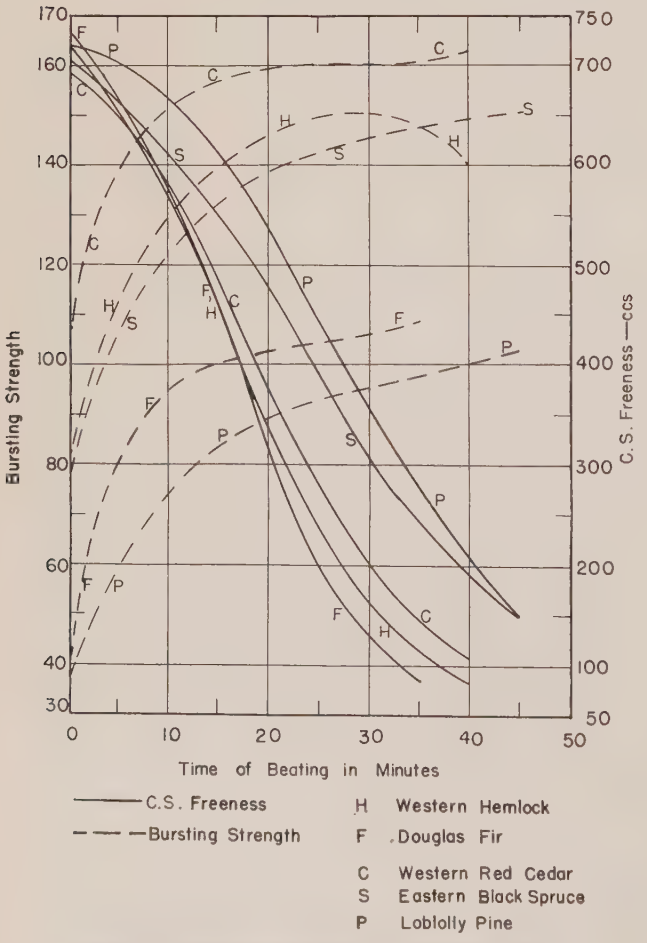


*Fig. 3. Bursting strengths of sulphite pulps*

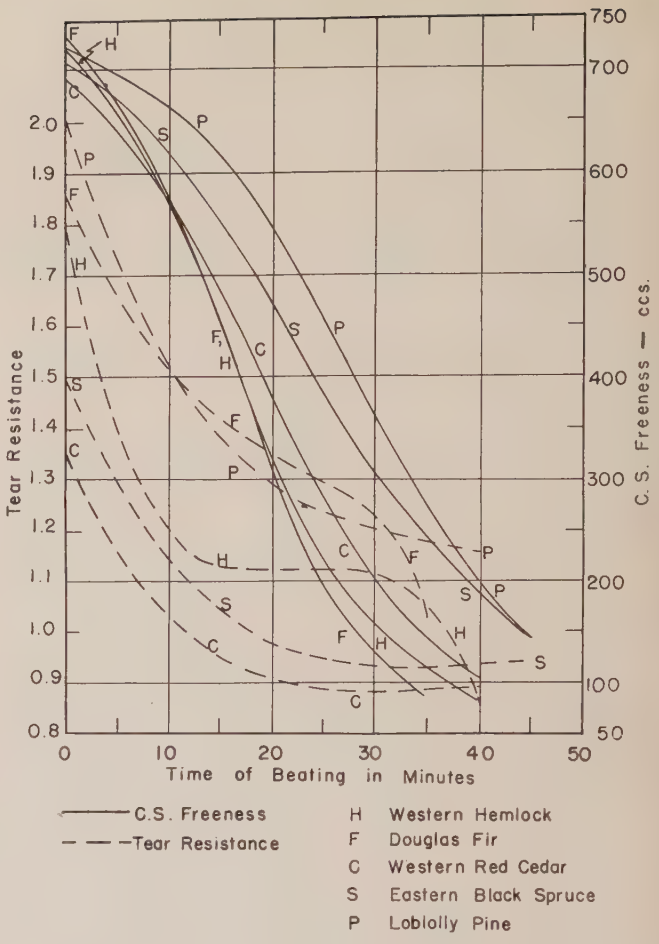


*Fig. 4. Tear resistances of sulphite pulps*

burst strengths far below the other three pulps. In tear resistance the Douglas fir pulp was easily the leader, followed by pine and hemlock, and lastly eastern spruce and cedar which had considerably lowered tear strength. In these tests, the beating time to 400 ml. Canadian Standard freeness showed no correlation with either strength property.

**Table VI. Physical Testing of the Sulphite Pulps**

| *Species* | *Western hemlock* | *Douglas fir* | *Cedar* | *Eastern spruce* | *Loblolly pine* |
|---|---|---|---|---|---|
| Burst, % | | | | | |
| at 500 ml. freeness | 137 | 99 | 156 | 137 | 92 |
| at 400 ml. freeness | 142 | 101 | 159 | 142 | 94 |
| Tear resistance | | | | | |
| at 500 ml. freeness | 1.13 | 1.44 | 0.96 | 0.99 | 1.26 |
| at 400 ml. freeness | 1.12 | 1.39 | 0.92 | 0.94 | 1.23 |
| Time to: | | | | | |
| 500 ml. freeness | 13.5 | 13 | 14.5 | 19 | 23 |
| 400 ml. freeness | 17 | 17 | 19 | 24 | 28 |

Mention should be made of pulp colors. Douglas fir, cedar, and loblolly pine are not typical sulphite woods and produced dark pulps as well as cooking with difficulty. Douglas fir was an olive color when wet which turned brown on drying; cedar was a light kraft color, and loblolly pine was a fairly dark gray. The color of the last pulp was due partly to some blue mold which had attacked the species in typical fashion.

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# The Characteristics of Unbleached Kraft Pulps from Western Hemlock, Douglas Fir, Western Red Cedar, Loblolly Pine, and Black Spruce

## *IV. The Specific Surface Areas of the Pulps and the Changes on Beating*

B. L. BROWNING and PHILIP S. BAKER

**When the specific surface areas and physical characteristics of the five pulps were measured after 5 and 50 minutes of beating, respectively, the pulps having the lowest bursting strength values were those showing the lowest specific surface areas. In fact, it was possible to arrange the five pulps in order of increasing surface for the same beating times and have the resulting order hold for increasing bursting strength values. Arranged in order of increasing specific surface and bursting strength, the pulps are: fir, pine, hemlock, spruce, and cedar. Both bursting and tensile strength values show a relationship to surface area independent of the pulp being tested, although the present data are insufficient to determine the exact relationships. It seems reasonable to conclude that one reason why both pine and fir pulps display low bursting strengths is the low specific surface areas shown by beaten pulps of those species.**

One possible means for evaluating or explaining the physical properties of pulps is the measurement of their specific surface areas. The specific surface of a pulp is defined as the exposed area in square centimeters per gram of moisture-free pulp; the determination provides a measure of the degree of fineness and/or fibrillation. The procedure consists in coating the surfaces of a pulp suspension with silver and then estimating the extent of silvered surface by measuring the rate of decomposition of hydrogen peroxide.

The early work on the method was done by Clark (*1*) and the method was improved by McEwen (*2*). In each case, the method was used substantially with unbeaten pulps. Although the extension of the method to cover the measurement of beaten pulps was still in its developmental stage at the time this work was undertaken, it appeared highly desirable to determine the significances of the determination in connection with kraft pulps.

Since the Clark-McEwen method in many uses gave erratic results with beaten pulps, a considerably modified version of their method was used in exploratory experiments on unbeaten samples of western hemlock, Douglas fir, and western red cedar unbleached kraft pulps. Since significant differences were indicated, beater runs were made and samples taken at five intervals of 5, 20, 35, 50, and 65 minutes for the measurement of surface area.

The technique used in the first series of experiments was as follows.

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The suspension from the beater was diluted to a suitable consistency with tap water and several aliquots were measured out. Consistency was determined by filtering off two of these samples on Jena 1-G-3 crucibles, removing the fibers as completely as possible from the crucibles, and drying and weighing them in tared weighing bottles.

For silvering, the aliquots were also filtered off on Jena 1-G-3 crucibles, the fibers were removed and suspended in 100 ml. of boiling dilute silvering solution (10 grams of silver nitrate per liter), and the silvering was carried out slightly below the boiling point for a time equivalent to twice the period required for the fibers to change color from red brown to black. At the end of this time, the fibers were filtered on a Jena 1-G-3 crucible, washed well, and rinsed into the reaction flask. One hundred milliliters

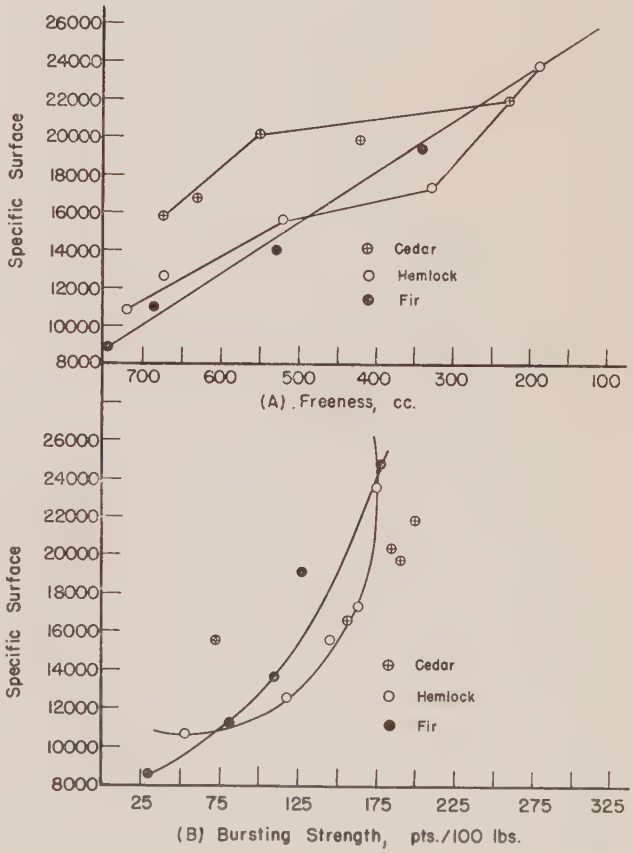


*Fig. 1. Relation between specific surface and (a) freeness and (b) bursting strength (data from Table I)*

Table I. Relation Between Beating Time, Specific Surface, and Pulp Strength of Fir, Hemlock, and Cedar Kraft Pulps Using the Original Method for Surface Area

| | Beating time, min. | Canadian freeness, ml. | Specific surface | Bursting strength, pt./100 lb. | Tear factor | Tensile strength, lb./in. | Stretch, % | Apparent density |
|---|---|---|---|---|---|---|---|---|
| Fir | 5 | 750 | 8,700 | 29 | 3.13 | 11.1 | 1.2 | 8.0 |
| | 20 | 685 | 11,125 | 81 | 3.96 | 25.1 | 2.2 | 10.5 |
| | 35 | 530 | 13,725 | 112 | 3.34 | 27.5 | 2.2 | 11.0 |
| | 50 | 340 | 19,275 | 127 | 2.82 | 32.9 | 2.6 | 11.5 |
| | 65 | 180 | 24,725 | 135 | 2.51 | 33.9 | 2.8 | 12.6 |
| Hemlock | 5 | 725 | 10,720 | 51 | 3.63 | 17.3 | 2.1 | 10.0 |
| | 20 | 675 | 12,475 | 114 | 2.32 | 33.0 | 2.7 | 11.5 |
| | 35 | 520 | 15,460 | 145 | 1.93 | 37.5 | 2.9 | 13.0 |
| | 50 | 330 | 17,150 | 162 | 1.66 | 40.6 | 3.4 | 13.5 |
| | 65 | 185 | 23,800 | 168 | 1.62 | 42.4 | 3.3 | 14.0 |
| Cedar | 5 | 675 | 15,740 | 72 | 3.99 | 18.0 | 2.7 | 10.5 |
| | 20 | 630 | 16,625 | 158 | 2.12 | 34.5 | 3.6 | 12.5 |
| | 35 | 550 | 20,160 | 184 | 1.92 | 38.1 | 4.0 | 13.5 |
| | 50 | 420 | 19,740 | 192 | 1.74 | 39.2 | 3.5 | 14.0 |
| | 65 | 225 | 21,960 | 201 | 1.54 | 44.0 | 4.1 | 14.5 |

of dilute borate buffer and 25 ml. of 0.1 *N* hydrogen peroxide were added. The catalytic decomposition was carried out for exactly 100 seconds after the addition of the hydrogen peroxide, and the temperature was recorded at the end of 50 seconds. At the end of the 100 seconds, the reaction was stopped by the addition of 15 ml. of 2 *N* sulphuric acid, the undecomposed hydrogen peroxide was titrated with 0.1 *N* potassium permanganate, and the surface area (S. A.) obtained from a calibration curve. The actual surface area in square centimeters per gram was obtained from the equation:

$$\text{Specific surface} = \frac{\text{S. A. of sample} \times 1000}{\text{Consistency (g./l.)} \times \text{ml. of sample}}$$

The data on specific surface, freeness, and the physical tests on handsheets will be found in Table I. For the first three beating periods, the surface areas increased regularly with decrease in freeness and tear, and increase in bursting and tensile strengths, stretch, and apparent density. The last two beating periods showed an apparent decrease in the rate of surface area increase with western hemlock and a cessation of that increase for western red cedar (Fig. 1). The explanation lies, in part at least, in the loss of fines during the filtering operations, particularly in the case of the cedar and fir. One cursory test with the cedar showed the loss to be as high as 16%. Contrary to this change in rate of formation of specific surface, the strength values follow their expected courses.

Further experiments were then carried out to standardize the method further and to eliminate its more evident weaknesses. In the final tests, distilled water replaced tap water, and the silvering was carried out by adding a concentrated silvering solution (100 grams of silver nitrate per liter) to an unfiltered boiling aliquot of the suspension. The silvered fibers were filtered on an asbestos-fitted Gooch crucible to prevent the loss of the fines. Consistencies were determined by filtering 500-ml. aliquots through asbestos-fitted Gooch crucibles.

Beater tests were again made with the five pulps but, because of lack of time, only two points were taken for test—at 5 and 50-minute beating intervals. For the time being, it can be said that this method gives consistent results but much more remains to be done in the preparation of the fiber for silvering before the data can assume true fundamental significance.

The data for the two periods will be found in Table II.

These data are shown in Fig. 2. Lines have been drawn between the two points in the specific surface vs. freeness figure; all bursting-strength values have been included in the specific surface vs. bursting-strength figure. The freeness lines arrange themselves in the same order as the bursting strengths of the pulps—the pulps showing low specific surfaces at a given freeness have the lowest bursting strengths at those freenesses. However, when the bursting strengths are plotted against the specific surfaces, some regularity will be noted except for the two values

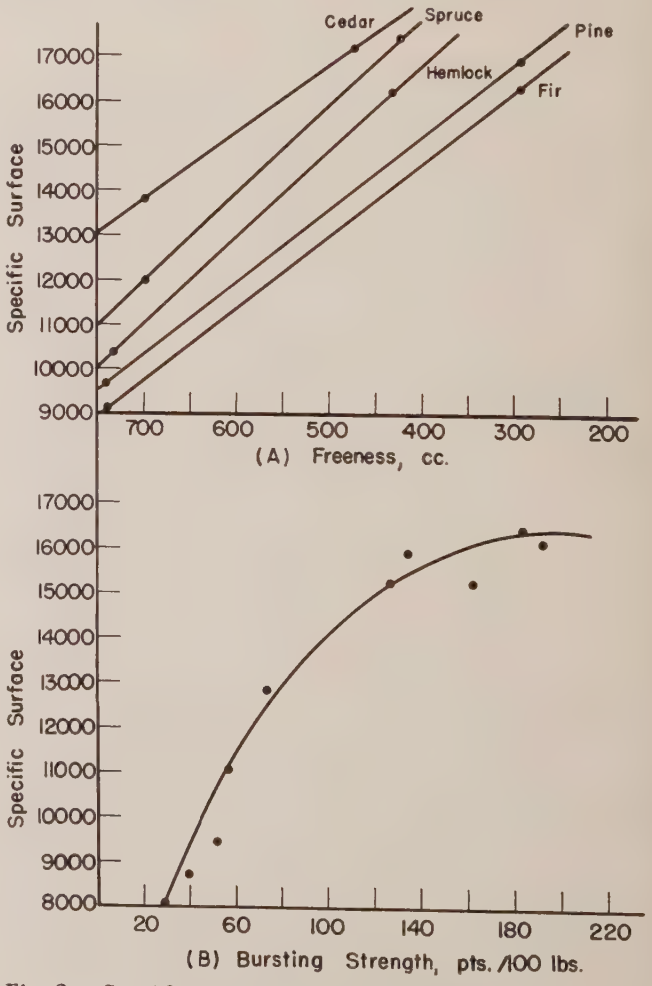


*Fig. 2. Specific surface vs. (a) freeness and (b) bursting strength (data from Table II)*

Table II. Strength Data and Specific Surface Data Obtained by Modified[a] Method

| Pulp | Time, min. | Specific surface | Canadian freeness, ml. | Bursting sternath, pt./100 lb. | Tensile strength lb./in. | Tear factor |
|---|---|---|---|---|---|---|
| Loblolly pine | 5 | 9,700 | 740 | 39 | .. | 3.73 |
| | 50 | 16,900 | 295 | 135 | .. | 2.05 |
| Spruce | 5 | 12,000 | 700 | 57 | .. | 4.08 |
| | 50 | 17,400 | 425 | 184 | .. | 1.46 |
| Red cedar | 5 | 13,800 | 700 | 72 | 18.0 | 3.99 |
| | 50 | 17,100 | 475 | 192 | 39.2 | 1.79 |
| Western hemlock | 5 | 10,400 | 735 | 51 | 17.3 | 3.63 |
| | 50 | 16,200 | 435 | 162 | 40.6 | 1.66 |
| Douglas fir | 5 | 9,100 | 740 | 29 | 11.1 | 3.13 |
| | 50 | 16,200 | 295 | 127 | 32.9 | 2.82 |

[a] Specific surface determination modified as indicated on page 100.

for western hemlock which are below the line. There would appear to be some relationship between bursting strength and surface area; in fact, when the limitations in both tests are considered, the relationship appears even more significant. Just what the significance is can be determined only through a more extended study. Similarly, tensile values have some relationship to the specific surfaces (only six values are available). The tear factors for high-freeness values vary widely—at the lower freeness values, the tear factors (except the value for the hemlock pulp) show a general linear relationship.

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2. McEwen, John M. "A Study of the Action of Silvered Pulps on Hydrogen Peroxide as a Means of Determining Their Specific Surface." Master's Dissertation, The Institute of Paper Chemistry, Appleton, Wis., 1941.



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# The Characteristics of Unbleached Kraft Pulps from Western Hemlock, Douglas Fir, Western Red Cedar, Loblolly Pine, and Black Spruce

## *V. A Study of the Distribution of the Degree of Polymerization of the Cellulose in the Kraft Pulps*


EMIL HEUSER, WILLIAM SHOCKLEY, and R. KJELLGREN



**The average chain length and the chain-length distribution have been determined for western hemlock, western red cedar, Douglas fir, eastern spruce, and loblolly pine kraft pulps by methods involving fractionation by solvent extraction. The results show that eastern spruce pulp closely resembles Douglas fir pulp, and that loblolly pine pulp is similar to those from western red cedar and western hemlock. The limitations of the method of fractionation by solvent extraction are pointed out. More recent work favors the fractional precipitation technique, particularly in the upper D.P. regions.**


It has been shown that the physical properties of cellulose derivatives are related to the degree of polymerization and the distribution of the degree of polymerization. Likewise, in the case of pulps of the same type from the same wood, a high average degree of polymerization, as measured by the cuprammonium viscosity, appears to be associated with a pulp of high strength, whereas a pulp of low viscosity will show less desirable strength properties. Viscosity is a measure of the average degree of polymerization; however, it does not establish the range or the distribution.


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It might be reasoned that the different pulps would show significant differences in the distribution of the cellulose molecular sizes. One pulp might contain a fairly large percentage in the low molecular range—these would swell more easily and serve to act as cementing material in the sheet. Another pulp might contain much of the high molecular weight material and, during beating, might behave more like a chemically refined pulp of high alpha-cellulose content.

The method used for the determination of the distribution of the degrees of polymerization was as follows: Pulps were converted into their nitrates by a procedure (using a mixture of nitric and phosphoric acids in the presence of phosphorus pentoxide) which causes practically no degradation of the cellulose. The nitrates were dissolved in acetone and subjected to specific viscosity determinations. The viscosity data were used for the calculation of the molecular weights on the basis of the Staudinger function. In this way the average molecular weights and, by dividing them by 162 (the molecular weight of the anhydroglucose unit), the average degrees of polymerization (D.P.'s) were obtained.

In order to determine the chain-length frequency

**Table I. Average Chain Length and Chain-Length Distribution of Western Hemlock Kraft Pulp**

| Fraction | Weight, % 1 | Weight, % 2 | Weight, % Av. | D.P. 1 | D.P. 2 | D.P. Av. | Calculated D.P. × % 1 | Calculated D.P. × % 2 | D.P. (Corr.), Av. |
|---|---|---|---|---|---|---|---|---|---|
| I | 16.03 | 15.13 | 15.58 | 217 | 135 | 176 | 34.79 | 20.43 | 197 |
| II | 9.82 | 6.79 | 8.40 | 1190 | 1005 | 1098 | 116.86 | 68.24 | 1230 |
| III | 8.05 | 7.12 | 7.58 | 1423 | 1362 | 1398 | 114.55 | 96.97 | 1566 |
| IV | 8.62 | 9.21 | 8.92 | 1450 | 1402 | 1426 | 124.99 | 129.12 | 1597 |
| V | 12.40 | 13.59 | 13.00 | 1450 | 1480 | 1465 | 179.80 | 201.13 | 1641 |
| VI | 6.55 | 8.84 | 7.69 | 1495 | 1510 | 1502 | 97.92 | 133.48 | 1682 |
| VII | 5.26 | 6.01 | 5.64 | 1500 | 1692 | 1596 | 78.90 | 101.69 | 1788 |
| VIII | 33.27 | 33.31 | 33.29 | 1615 | 1830 | 1723 | 537.31 | 609.57 | 1930 |
| | | | | | | | 1285 | 1361 | |

Average D.P. of unfractionated nitrate—1481 (1500 and 1462)
Average D.P. calculated from fractions—1323 (1285 and 1361)
Corrected D.P. values obtained by using a factor of 1481/1323 = 1.12
Yield on nitration—171.0% (170.5 and 171.5%)

Solvent Mixtures Used for Fractionation

| Fraction | Alcohol | Ethyl acetate | Fraction | Alcohol | Ethyl acetate |
|---|---|---|---|---|---|
| I | 10 | 12 | V | 10 | 22 |
| II | 10 | 16 | VI | 10 | 24 |
| III | 10 | 18 | VII | 10 | 26 |
| IV | 10 | 20 | VIII | Pure solvent | |

**Table II. Average Chain Length and Chain-Length Distribution of Western Red Cedar Kraft Pulp**

| Fraction | Weight, % 1 | Weight, % 2 | Weight, % Av. | D.P. 1 | D.P. 2 | D.P. Av. | Calculated D.P. × % 1 | Calculated D.P. × % 2 | D.P. (Corr.), Av. |
|---|---|---|---|---|---|---|---|---|---|
| I | 9.87 | 10.39 | 10.13 | 110 | 131 | 120 | 10.86 | 13.61 | 130 |
| II | 2.55 | 3.77 | 3.16 | 544 | 872 | 708 | 13.87 | 32.87 | 765 |
| III | 2.27 | 4.33 | 3.30 | 1056 | 1235 | 1146 | 23.97 | 53.48 | 1238 |
| IV | 4.50 | 4.50 | 4.50 | 1459 | 1591 | 1525 | 65.66 | 71.60 | 1647 |
| V | 9.49 | 11.56 | 10.52 | 1297 | 1498 | 1398 | 123.09 | 173.17 | 1510 |
| VI | 7.92 | 7.63 | 7.78 | 1596 | 1630 | 1613 | 126.40 | 124.37 | 1742 |
| VII | 8.98 | 10.92 | 9.95 | 1722 | 1761 | 1742 | 154.64 | 192.30 | 1881 |
| VIII | 16.55 | 6.61 | 11.58 | 1878 | 1818 | 1848 | 310.81 | 120.17 | 1996 |
| IX | 37.87 | 40.29 | 39.08 | 1788 | 1751 | 1768 | 675.60 | 705.48 | 1909 |
| | | | | | | | 1505 | 1487 | |

Average D.P. of unfractionated nitrate—1615 (1625 and 1605)
Average D.P. calculated from fractions—1496 (1505 and 1487)
Corrected D.P. values obtained by using a factor of 1615/1496 = 1.08
Yield on nitration—171.0% (171.0 and 171.0%)

Solvent Mixtures Used for Fractionation

| Fraction | Alcohol | Ethyl acetate | Fraction | Alcohol | Ethyl acetate |
|---|---|---|---|---|---|
| I | 10 | 12 | VI | 10 | 24 |
| II | 10 | 16 | VII | 10 | 26 |
| III | 10 | 18 | VIII | 10 | 28 |
| IV | 10 | 20 | IX | Pure solvent | |
| V | 10 | 22 | | | |

**Table III. Average Chain Length and Chain-Length Distribution of Douglas Fir Kraft Pulp**

| Fraction | Weight, % 1 | Weight, % 2 | Weight, % Av. | D.P. 1 | D.P. 2 | D.P. Av. | Calculated D.P. × % 1 | Calculated D.P. × % 2 | D.P. (Corr.), Av. |
|---|---|---|---|---|---|---|---|---|---|
| I | 13.10 | 12.73 | 12.91 | 125 | 97 | 111 | 16.38 | 12.34 | 121 |
| II | 5.22 | 4.28 | 4.75 | 1024 | 906 | 965 | 53.45 | 38.77 | 1052 |
| III | 7.07 | 5.19 | 6.13 | 1499 | 1445 | 1472 | 105.98 | 75.00 | 1604 |
| IV | 7.71 | 6.67 | 7.19 | 1700 | 1485 | 1598 | 131.07 | 99.05 | 1742 |
| V | 8.56 | 7.39 | 7.98 | 1595 | 1851 | 1723 | 136.53 | 136.79 | 1878 |
| VI | 17.75 | 18.88 | 18.32 | 1724 | 1650 | 1687 | 306.01 | 311.52 | 1839 |
| VII | 6.36 | 8.50 | 7.43 | 1831 | 1710 | 1771 | 116.45 | 145.35 | 1930 |
| VIII | 8.86 | 10.06 | 9.46 | 1895 | 1983 | 1939 | 167.90 | 199.49 | 2114 |
| IX | 25.37 | 26.30 | 25.83 | 2078 | 2032 | 2055 | 527.18 | 534.42 | 2240 |
| | | | | | | | 1561 | 1553 | |

Average D.P. of unfractionated nitrate—1695 (1690 and 1700)
Average D.P. calculated from fractions—1557 (1561 and 1553)
Corrected D.P. values obtained by using a factor of 1695/1557 = 1.09
Yield on nitration—170.2% (169.5 and 172.0%)

Solvent Mixtures Used for Fractionation

| Fraction | Alcohol | Ethyl acetate | Fraction | Alcohol | Ethyl acetate |
|---|---|---|---|---|---|
| I | 10 | 12 | VI | 10 | 24 |
| II | 10 | 16 | VII | 10 | 26 |
| III | 10 | 18 | VIII | 10 | 28 |
| IV | 10 | 20 | IX | Pure solvent | |
| V | 10 | 22 | | | |

**Table IV. Chain-Length Distribution of Eastern Spruce Unbleached Kraft Pulp**

| Fraction | Weight, % | | | D.P. | | | Calculated D.P. × % | | D.P. (Corr.), Av. |
|---|---|---|---|---|---|---|---|---|---|
| | 1 | 2 | Av. | 1 | 2 | Av. | 1 | 2 | |
| I | 15.46 | 15.38 | 15.42 | 107 | 100 | 104 | 16.54 | 15.38 | 116 |
| II | 4.77 | 3.25 | 4.01 | 990 | 705 | 848 | 47.22 | 22.91 | 950 |
| III | 4.65 | 3.25 | 3.95 | 1245 | 957 | 1101 | 57.89 | 31.10 | 1233 |
| IV | 6.08 | 5.00 | 5.54 | 1410 | 1350 | 1380 | 85.73 | 67.50 | 1546 |
| V | 6.37 | 4.65 | 5.51 | 1562 | 1340 | 1451 | 99.50 | 62.31 | 1625 |
| VI | 5.59 | 3.95 | 4.77 | 1585 | 1455 | 1520 | 88.60 | 57.47 | 1702 |
| VII | 7.47 | 6.58 | 7.02 | 1608 | 1528 | 1568 | 120.12 | 100.54 | 1756 |
| VIII | 13.08 | 14.54 | 13.81 | 1765 | 1688 | 1728 | 230.86 | 245.44 | 1935 |
| IX | 13.39 | 15.67 | 14.53 | 1905 | 1835 | 1870 | 255.08 | 287.54 | 2094 |
| X | 23.14 | 27.73 | 25.44 | 1955 | 1985 | 1970 | 452.39 | 550.44 | 2206 |
| | | | | | | | 1454 | 1441 | |
| | | | | | | | | Av. = 1448 | |

Average D.P. of unfractionated nitrate—1619 (1610 and 1628)
Average D.P. calculated from fractions—1448 (1454 and 1441)
D.P. values were corrected by using a factor of = 1.12
Yield on nitration—167.9% (169.2 and 166.5%)

Solvent Mixtures Used for Fractionation

| Fraction | Alcohol | Ethyl acetate | Fraction | Alcohol | Ethyl acetate |
|---|---|---|---|---|---|
| I | 10 | 12 | VI | 10 | 24 |
| II | 10 | 16 | VII | 10 | 26 |
| III | 10 | 18 | VIII | 10 | 28 |
| IV | 10 | 20 | IX | 10 | 30 |
| V | 10 | 22 | X | Pure solvent | |

distribution of the pulp, the nitrates were subjected to fractional solution (extraction) with a mixture of 95% ethanol as nonsolvent and ethyl acetate as solvent, keeping the volume of the nonsolvent constant and adding increasing quantities of the solvent. In this way the short-chain material is extracted first. As the amount of solvent is increased, increasingly longer chains are removed. Thus, seven to eight fractions were obtained with hemlock, cedar, and fir, and nine fractions were obtained with pine and spruce. A ninth fraction, representing the remaining residue, was obtained by using the solvent alone (that is, without alcohol being present) in the case of the first three pulps, and a tenth fraction in the case of the last two, the difference being that, with the pine and spruce pulps, one more fraction was added by increasing the proportion of the solvent in the solvent-nonsolvent mixture from 28 to 30 parts before the pure solvent was used. This was done in the hope that the large fraction of highest molecular weight might be further subdivided. However, better subdivision was achieved only to a limited extent.

After the various extractions, the fractions were obtained by evaporating the solvent. The residues were dried and weighed and the weights were calculated to percentages of the weight of the unfractionated nitrate. The fractions were then subjected to viscosity measurements in acetone and the viscosity data were converted into molecular weight and D.P.'s as described above.

**Table V. Chain-Length Distribution of Loblolly Pine Unbleached Kraft Pulp**

| Fraction | Weight, % | | | D.P. | | | Calculated D.P. × % | | D.P. (Corr.), Av. |
|---|---|---|---|---|---|---|---|---|---|
| | 1 | 2 | Av. | 1 | 2 | Av. | 1 | 2 | |
| I | 9.91 | 10.40 | 10.15 | 91 | 81 | 86 | 9.02 | 8.42 | 96 |
| II | 2.61 | 2.45 | 2.53 | 535 | 560 | 547 | 13.96 | 13.72 | 612 |
| III | 3.07 | 2.92 | 3.00 | 806 | 882 | 844 | 24.74 | 25.75 | 945 |
| IV | 3.18 | 2.98 | 3.08 | 1090 | 1195 | 1143 | 34.66 | 35.61 | 1280 |
| V | 4.15 | 3.13 | 3.64 | 1475 | 1268 | 1377 | 61.21 | 39.69 | 1542 |
| VI | 6.46 | 6.41 | 6.44 | 1428 | 1451 | 1440 | 92.25 | 93.00 | 1613 |
| VII | 3.43 | 3.31 | 3.37 | 1465 | 1502 | 1484 | 50.25 | 49.72 | 1662 |
| VIII | 11.24 | 10.51 | 10.87 | 1700 | 1510 | 1605 | 191.08 | 158.70 | 1798 |
| IX | 41.14 | 42.58 | 41.86 | 1762 | 1790 | 1776 | 724.89 | 762.18 | 1989 |
| X | 14.81 | 15.31 | 15.06 | 1828 | 1905 | 1867 | 270.73 | 291.66 | 2091 |
| | | | | | | | 1473 | 1478 | |
| | | | | | | | | Av. = 1476 | |

Average D.P. of unfractionated nitrate—1659 (1648 and 1670)
Average D.P. calculated from fractions—1476 (1473 and 1478)
D.P. values were corrected by using a factor of = 1.12
Yield on nitration—172.5% (172.0 and 173.0%)

Solvent Mixtures Used for Fractionation

| Fraction | Alcohol | Ethyl acetate | Fraction | Alcohol | Ethyl acetate |
|---|---|---|---|---|---|
| I | 10 | 12 | VI | 10 | 24 |
| II | 10 | 16 | VII | 10 | 26 |
| III | 10 | 18 | VIII | 10 | 28 |
| IV | 10 | 20 | IX | 10 | 30 |
| V | 10 | 22 | X | Pure solvent | |

One way to picture the chain-length distribution is to plot the percentage weights of the fractions against the D.P.'s This procedure results in a step curve which shows the percentage weight of the short, medium, and long chains in the pulp [see Figs. 1–5]. In Figs. 6 and 7 the results are represented as differential weight distribution curves. These curves were obtained by point-by-point differentiation of the smooth curves into which the step curves of Figs. 1–5 were converted by interpolation. In the graphic representation, $m(P) \times 10^4$ is plotted against the degree of polymerization. The term $m(P) \times 10^4$ represents the frequency of occurrence of a given molecular weight increment. This frequency, when multiplied by the increment, gives the total weight of the chains within

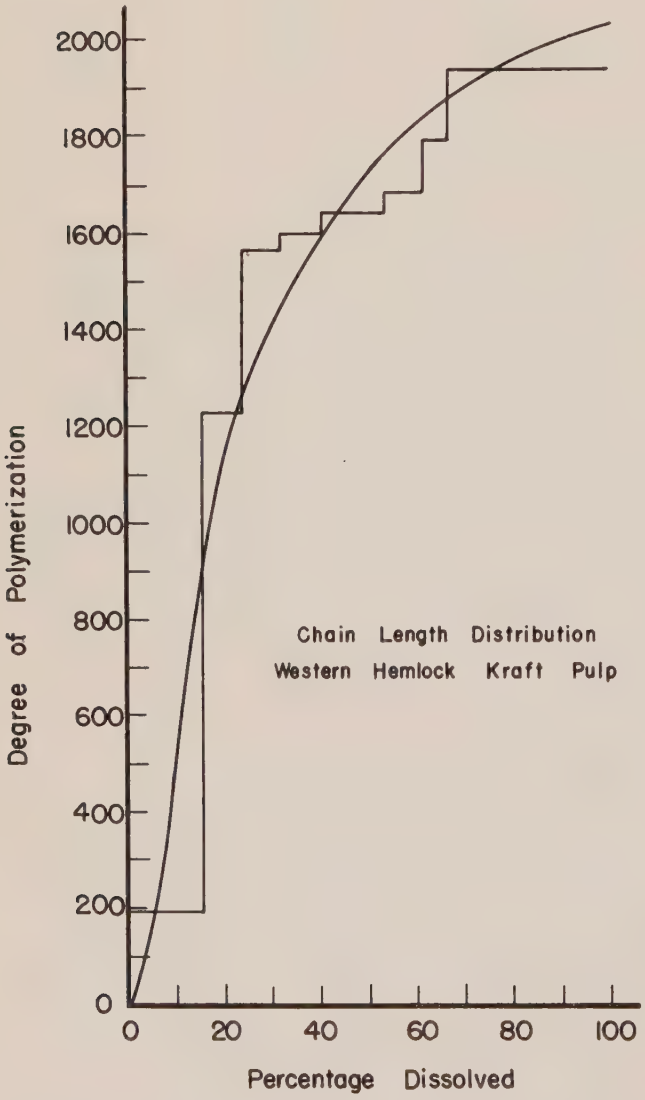


*Fig. 1. Chain-length distribution of western hemlock kraft pulp*

the increment and the function $[m(P)]$, when integrated between given D.P. limits, gives the total weight of polymer within those limits. The details of the investigation of the five pulps are shown in Tables I–V.

## DISCUSSION

### *1. Western Hemlock Pulp*

The actually determined average D.P. of 1481 would appear to be within the usual range for unbleached kraft pulps.

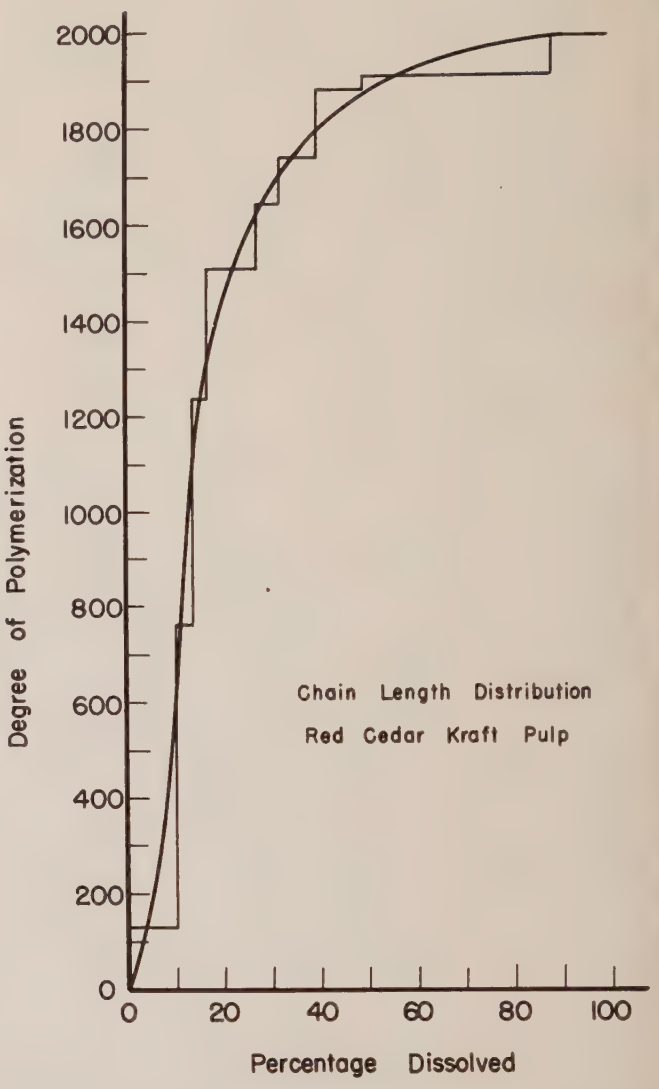


*Fig. 2. Chain-length distribution of western red cedar kraft pulp*

The interpretation of the chain-length distribution data (Table I, columns 4 and 10) is facilitated by grouping fractions of similar D.P.'s together. Thus, the values shown in Table VI are obtained:

**Table VI**

| | % | D.P. |
|---|---|---|
| 1. | 15.58 | 197 |
| 2. | 8.40 | 1230 |
| 3. | 42.83 | 1566–1788 |
| 4. | 33.29 | 1930 |
| | 100.10 | |

It is seen from these data that the average D.P. of this pulp would have been higher, if there had not been present an appreciable amount (15 to 16%) of rather

short-chain material (D.P. 197). A further depression was caused by the presence of about 8.5% of medium-chain material (D.P. 1230). Of the long-chain material, an appreciable amount (about 43%) would appear to be rather uniform, being within the relatively narrow range of 1566 to 1788. Another appreciable

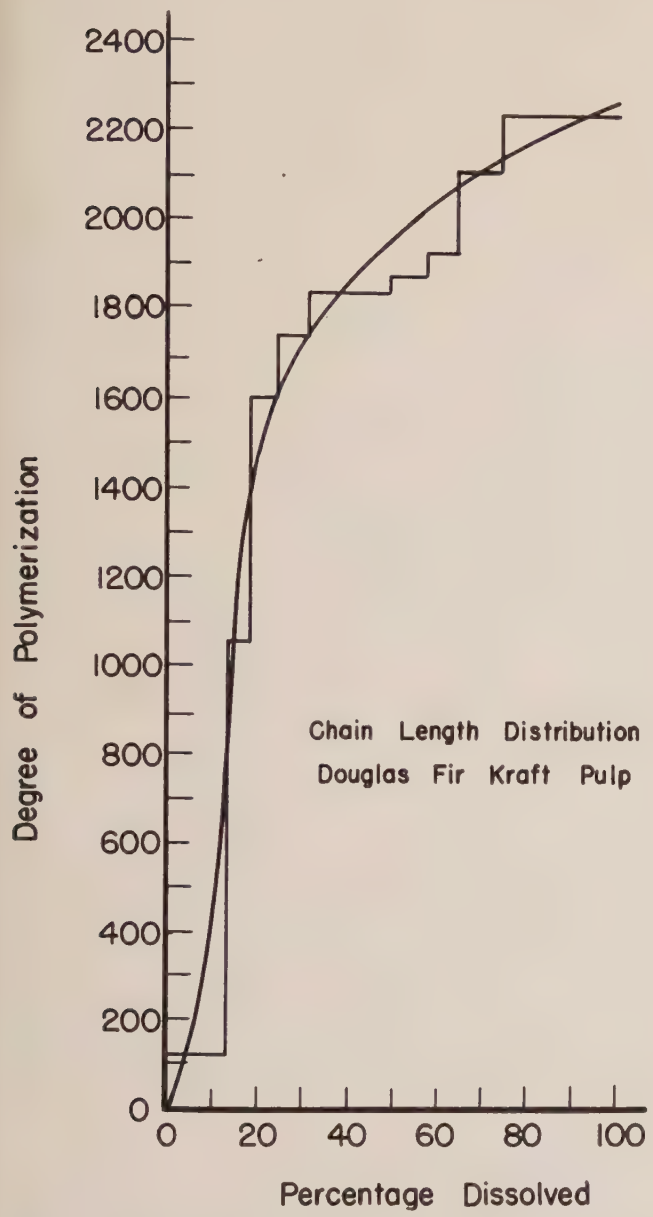


*Fig. 3. Chain-length distribution of Douglas fir kraft pulp*

amount (about 33%) reached a rather high level—i.e., 1930. However, it is probable that this last fraction contained portions of even higher D.P. (as well as material of a D.P. lower than 1930). On the basis of this interpretation, this pulp would appear to be of a medium degree of uniformity.

### 2. *Western Red Cedar Pulp*

The average D.P. of western red cedar pulp (1615) is higher than that of the previous pulp (1481), and its higher average is reflected in its chain-length distribution. Again, the results of fractions of similar D.P.'s may be grouped together:

**Table VII**

| | % | D.P. |
|---|---|---|
| 1. | 10.13 | 130 |
| 2. | 3.16 | 765 |
| 3. | 3.30 | 1238 |
| 4. | 22.80 | 1510–1742 |
| 5. | 60.61 | 1881–1996 |

It is seen that the higher average D.P. of this pulp is in line with a shift of the long-chain material to a higher level (group 5) which, in addition, comprises a larger percentage than that of the highest level of the western hemlock pulp (see group 4 of Table VI).

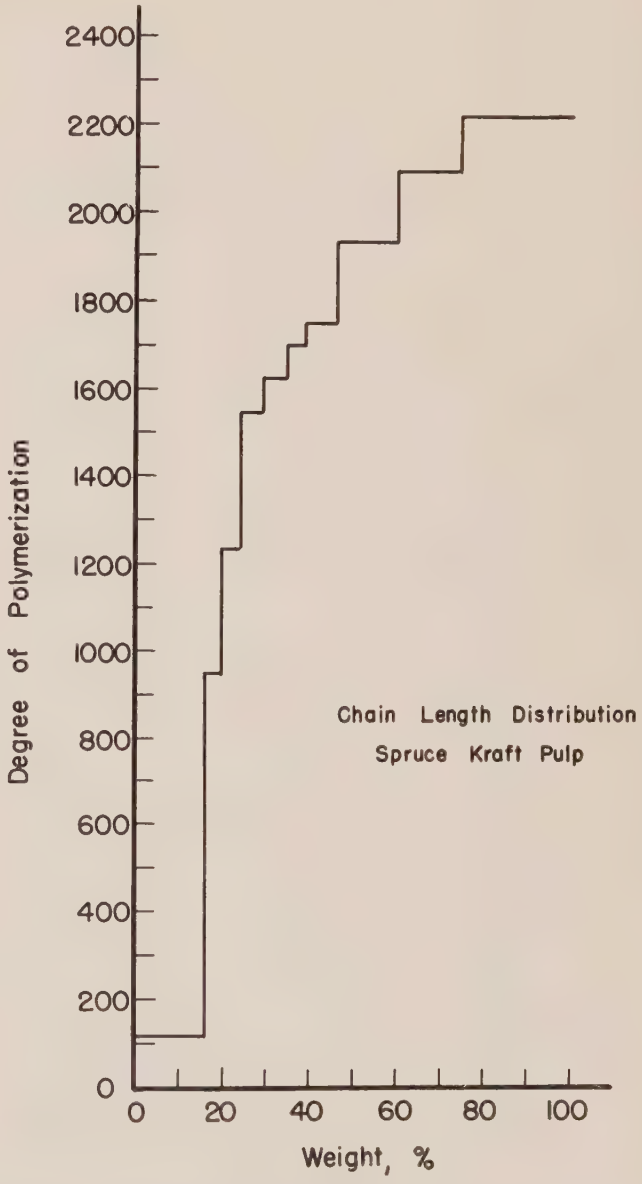


*Fig. 4. Chain-length distribution of eastern spruce unbleached kraft pulp*

Accordingly, the percentage of group 4 in the present pulp was smaller. It is of interest to note that this pulp also contained certain, though slightly smaller, amounts of very short-chain material (even shorter than that present in the previous pulp) and of medium-

chain material (groups 1 and 3). In addition, there was a small amount of material ranging between the very short and the medium materials (group 2). Although an appreciable percentage of this pulp ranges between rather narrow limits (group 5), the rest would seem to be somewhat more heterogeneous than that found for the western hemlock pulp; however, the difference in the over-all picture of the two pulps must be regarded as small. Thus, both pulps would appear to be of rather similar type.

It should be added that the western red cedar pulp (see Table II) was divided into nine instead of eight fractions. This was done because there was still a considerable residue after the seventh fraction had

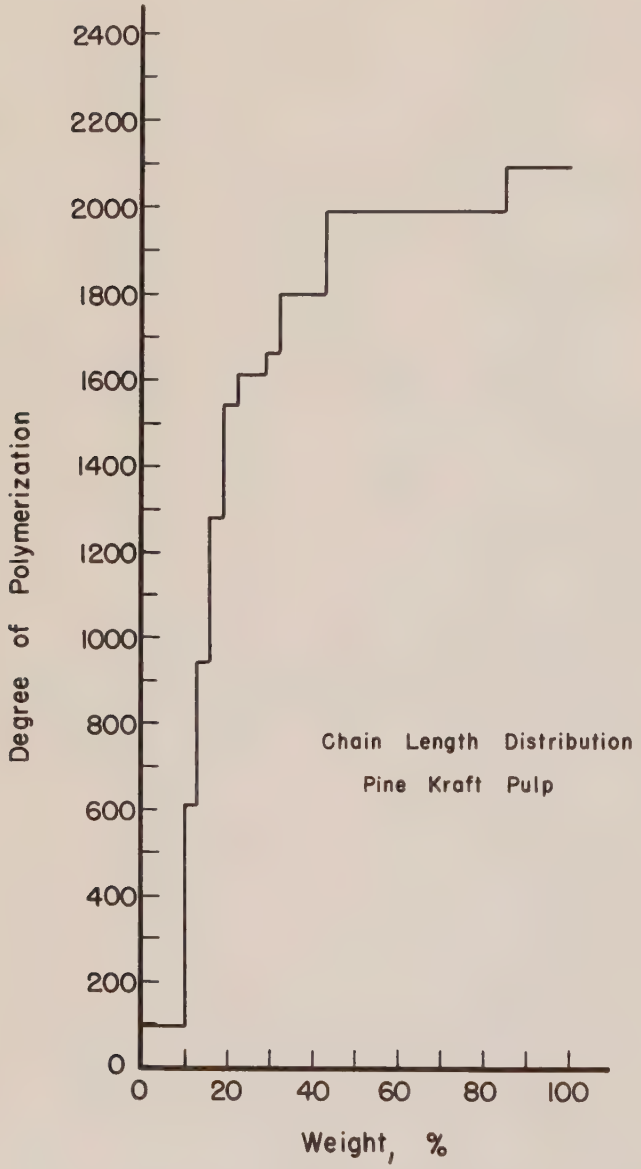


*Fig. 5. Chain-length distribution of loblolly pine unbleached kraft pulp*

been extracted. However, no further subdivision was obtained by the use of the 10–28 mixture; in fact, the seventh, eighth, and ninth fractions were all rather close together.

### 3. *Douglas Fir Pulp*

Douglas fir pulp had the highest average D.P. (1695) and the chain-length distribution data (again from nine instead of eight fractions) show that the long-chain material was at a considerably higher level and that a higher percentage of this was present than in the other two pulps. This is also seen when similar fractions are grouped together.

**Table VIII**

| | % | D.P. |
|---|---|---|
| 1. | 12.91 | 121 |
| 2. | 4.75 | 1052 |
| 3. | 13.32 | 1604–1742 |
| 4. | 33.73 | 1839–1930 |
| 5. | 35.29 | 2114–2240 |

Whereas the western red cedar pulp contained about 60% of material ranging from 1881 to 1926, the Douglas fir pulp contained about 69% of material between 1839 and 2240. In addition, there was less of the long-chain material of the D.P. range of 1604 to 1742, which is considered as group 3 (compare with group 4 of Table VII). The presence of medium- and very short-chain materials (groups 2 and 1) would seem to simulate the situation encountered in the red cedar and hemlock pulps.

On the whole, the Douglas fir pulp appears to be more heterogeneous than the other two pulps from western woods. This might be the result of a milder cook or of a greater resistance of Douglas firwood to pulping, as compared with the other two woods. Whatever the reason, the chain-length distribution of the carbohydrate material, as it originally existed in the Douglas firwood and which may reasonably be assumed to be very nonuniform, would seem to be more preserved than in the other two pulps which show the homogenizing influence of the pulping procedure to a greater extent. Although still unknown counteracting influences might have been operative, its chain-length distribution would lead us to expect that the Douglas fir pulp is a stronger pulp than the two others. On the same basis, the latter two would be expected to show similar strength properties, although slightly higher values might be found for the western red cedar than for the western hemlock pulp.

### 4. *Eastern Spruce Pulp*

The results for eastern spruce pulp are shown in Table IV.

The average D.P. of the eastern spruce pulp (1619) is somewhat closer to that of the western red cedar pulp (1615) than to that of the Douglas fir pulp (1695). However, its chain-length distribution is more comparable with that of the latter.

This is seen when fractions of similar D.P. are grouped together.

The somewhat lower average D.P. of the eastern spruce pulp, as compared with that of the Douglas fir pulp, obviously results from the presence in the former of a somewhat greater percentage of low D.P. material (groups 1 and 2). In addition, there are a few per cent of medium D.P. material (group 3) which was not found in the Douglas fir pulp.

This interpretation seems to be confirmed by the chain-length distribution diagrams (Fig. 4) and by the differential weight distribution curves (Fig. 7). The differential weight distribution curves for the western

**Table IX**

| | % | D.P. |
|---|---|---|
| 1. | 15.42 | 116 |
| 2. | 4.01 | 950 |
| 3. | 3.95 | 1233 |
| 4. | 15.82 | 1546–1702 |
| 5. | 20.83 | 1756–1935 |
| 6. | 39.97 | 2094–2206 |

wood pulps are shown in Fig. 6. However, in comparing curve 1 of Fig. 7 with curve 3 of Fig. 6, it will be noticed that the first maximum ("hump") shown by the eastern spruce pulp is less pronounced than that shown by the Douglas fir pulp.

### 5. *Loblolly Pine Pulp*

The results for the loblolly pine pulp are shown in Table V.

The yield on nitration (172.5%) was again in agreement with that found for the previously investigated pulps.. Although its average D.P. (1659) is somewhat closer to that of the Douglas fir pulp (1695) than to that of the red cedar pulp (1615), its chain-length distribution is more comparable with that of the latter.

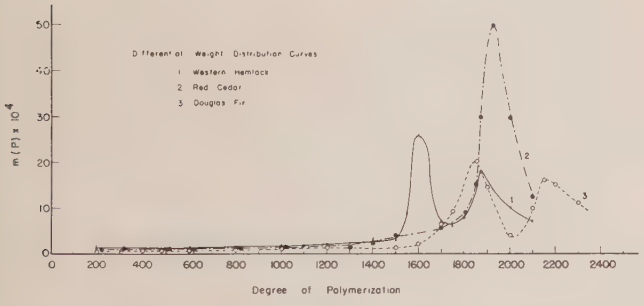


***Fig. 6. Differential weight distribution curves of (1) western hemlock kraft pulp, (2) western red cedar kraft pulp, and (3) Douglas fir kraft pulp***

This is seen from a comparison of the grouping of fractions of similar D.P. The same similarity between the two pulps is revealed in their chain-length distribution diagrams (Fig. 5).

**Table X. Percentages and D.P. Ranges of Five Kraft Pulps**

| Pulps | *Short-chain material* % | D.P. | *Long-chain material* % | D.P. | *Medium-chain material,* % |
|---|---|---|---|---|---|
| Western hemlock | 16 | 200 | 76 | 1560–1930 | 8 |
| Western red cedar | 10 | 130 | 83 | 1510–1946 | 7 |
| Loblolly pine | 10 | 96 | 81 | 1542–2091 | 9 |
| Douglas fir | 13 | 121 | 82 | 1604–2240 | 5 |
| Eastern spruce | 15.5 | 116 | 77 | 1546–2206 | 7.5 |

From a comparison of the five pulps on the basis of the step curves, it would seem that the eastern spruce pulp could be classified with the Douglas fir pulp, whereas the loblolly pine pulp would seem to be closer to the western red cedar and the western hemlock pulps. A characteristic of all five pulps is that they contain similar relatively small amounts of very short-chain material and similar large amounts of long-chain material, the remainder ranging between the two extremes (see Table X).

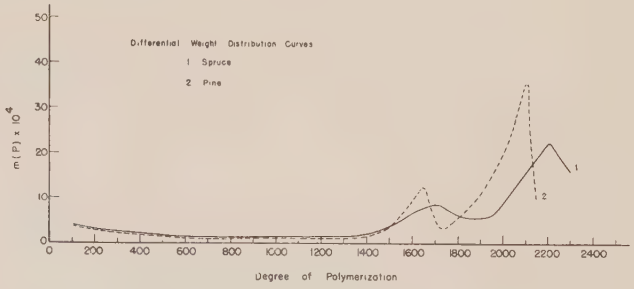


***Fig. 7. Differential weight distribution curves of (1) eastern spruce unbleached kraft pulp and (2) loblolly pine unbleached kraft pulp***

The results of this study lead to the conclusion that the method of fractional solution has its limitations and that, for a more thorough separation of the high molecular weight fractions of a given cellulosic material, other techniques will have to be used. It is apparent from the study that the distribution of molecular weight of the cellulose as measured by the method used does not play any primary part in the physical properties of those pulps.

The curves drawn to illustrate the differential weight distribution by degrees of polymerization show each pulp to have a different curve. The one pulp having an outstandingly different curve is that of western red cedar; 60% of this particular pulp is found in a narrow range of polymerization degree—between 1742 and 1996 (see Table VII).

On the basis of the viscosities of the unfractionated pulps and assuming that high viscosity means high strength, the pulps would be arranged in order of ascending strengths as follows: hemlock, cedar, spruce, pine, and fir. Actually, in terms of bursting strength, the order of rising progression is—fir, pine, hemlock, spruce, and cedar; a rise in tear is found in the order: spruce, cedar, hemlock, pine, and fir. The average degrees of polymerization of all these pulps are high and, with the possible exception of the much lower value for hemlock, are about the same; the futility of attempting to use viscosity alone as an index of strength in the comparison of pulps from different woods can be seen.

The particular value of pulp fractionation appears to be in the selection of pulps for rayon manufacture where random distribution of molecular sizes is undesirable. The determination of the unfractionated viscosity is valuable in the control of a process using the same raw material for the preparation of a specific type of bleached pulp.

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# The Characteristics of Unbleached Kraft Pulps from Western Hemlock, Douglas Fir, Western Red Cedar, Loblolly Pine, and Black Spruce

## *VI. The Orientation of Cellulose Chains in Pulp Fibers as Measured by X ray Diffraction*

G. L. CLARK

**An x ray diffraction method for evaluating quantitatively the type and degree of orientation of cellulose crystallites was applied on pulps of the five woods under study. A modification of the procedure gave significant results: the order of decreasing preferred orientation being Douglas fir, spruce, cedar, hemlock, and loblolly pine.**

In 1933 Sisson and Clark (*1*) proposed the experimental x ray diffraction method for evaluating quantitatively the type and degree of orientation of cellulose crystallites (and thereby the molecular chains) in various fibers. A single fiber, a parallel bundle of fibers, or a wood section is arranged so that a beam of x rays will pass through the specimen perpendicular to the fiber axis. The diffraction pattern is registered on a flat photographic film, usually at a distance of 5 cm. from the specimen. The method is based upon the assumption that the distribution of crystallites around the pencil of x rays is proportional to the distribution of intensity around the strong 002 diffraction ring of cellulose. Thus, a random orientation would be indicated by a continuous ring of uniform intensity, whereas a highly oriented state of the crystallites would be manifested by sharp intensity maxima on the equator of the ring (3 and 9 o'clock positions) with only faint evidences, if any, of the ring at the poles (12 and 6 o'clock positions). Intensity measurements are made with a microdensitometer equipped with a rotating stage. The distribution of crystallites as the percentage over each 5° angular range is calculated from these intensity values and the orientation is expressed by distribution curves which may be differentiated from one another by statistical methods. It was also shown (*1*) that the data thus obtained may be used to study the structure of various fibrous materials and to predict physical and chemical properties which are related to anisotropic structure, such as the tensile strength and elongation of cords.

Since 1933, this method has been used repeatedly and successfully for the comparison of various cellulose specimens. The newest application is to the comparison of wood pulps prepared from five kinds of wood.

Patterns were first made through a layer of the pulp with the fibers undisturbed and at random. This gave the typical cellulose pattern with complete rings exactly as expected. The visual inspection indicated very little difference from one pulp to another, although there was a quantitative difference in breadth, indicative of micellar size which varies to some extent from one species of wood to another. However, far more significant results were obtained by devising a technique whereby a small parallel bundle of individual fibers could be studied; this technique resulted in the patterns illustrated in Fig. 1. These are remarkably clear for wood cellulose and there is an amazing and distinct variation in the degree of preferred orientation in the individual fibers. As demonstrated many times, the higher the degree of preferred orientation, the greater the tensile strength but the smaller the pliability or elasticity, swelling properties, felting ability, etc. The degree of preferred orientation is indicated by the sharpness of the equatorial arcs of high intensity lying on the cellulose rings. In the order of decreasing preferred orientation, the series is very clearly as follows:

1. Douglas fir. This preferred orientation almost reaches that in ramie or flax and is exceptionally high for wood. There is a rather short intense band and, superposed upon this, almost a sharp spot right on the equator, which indicates a large proportion of the crystallites in almost perfect orientation parallel with each other and with respect to the fiber axis and the remaining crystallites in another group of preferred positions.

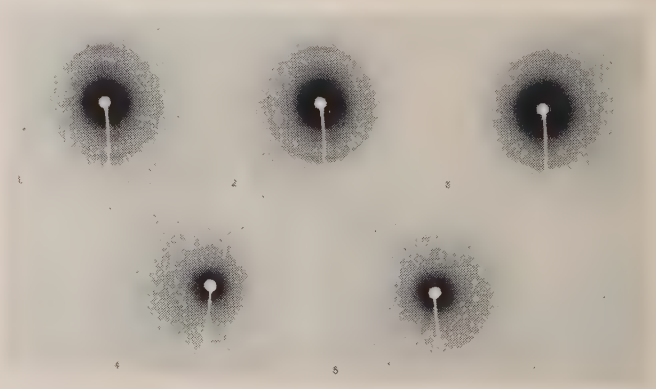

***Fig. 1. X ray diffraction patterns for wood pulp samples*** (1) Douglas fir; (2) spruce; (3) hemlock; (4) cedar; and (5) loblolly pine.

G. L. Clark, University of Illinois, Urbana, Ill.

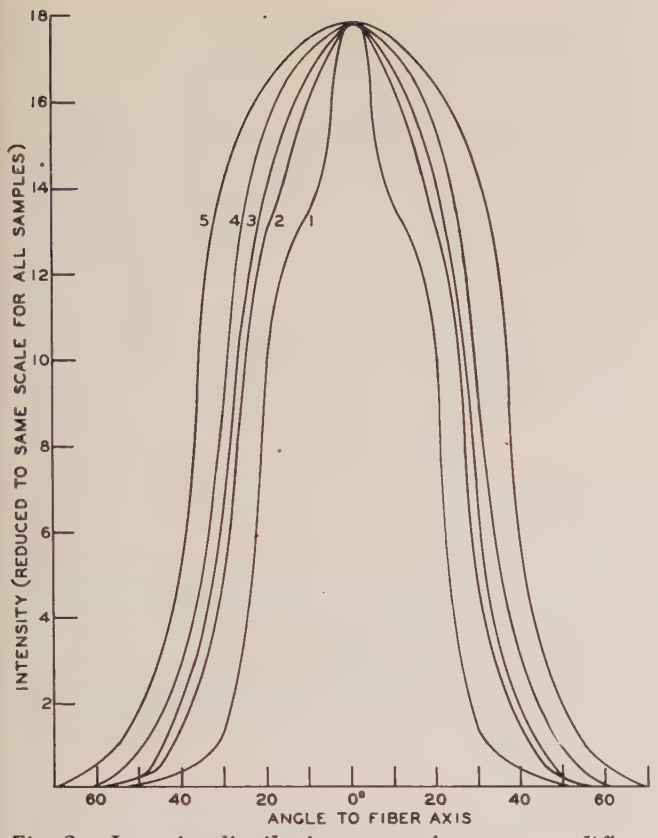


***Fig. 2. Intensity distribution curves for strongest diffraction rings on patterns in Fig. 1***

(1) Douglas fir; (2) spruce; (3) hemlock; (4) cedar; and (5) loblolly pine.

2. Spruce
3. Cedar
4. Hemlock
5. Loblolly pine

This last sample has only faint evidence of preferred orientation in the individual fibers. This is not a matter of variation between the fibers, but represents the structure within the individual fibers. It has occurred to us that this order may in some ways correspond with the rate of growth even under the same growing and weather conditions, as well as with other characteristics of each species of tree.

It is possible to make quantitative measurements with the microphotometer and to obtain a complete distribution curve of the orientation. In the present instances, the chords of the arcs on the patterns give a rapid approximate rating of preferred orientation with the following results:

| | |
|---|---|
| Douglas fir | 0.8 and 1.7 cm. |
| Spruce | 2.0 cm. |
| Cedar | 2.3 cm. |
| Hemlock | 2.4 cm. |
| Loblolly pine | 3.0 cm. |

The differences between Douglas fir, three intermediate pulps, and loblolly pine can be clearly seen. Douglas fir shows the shortest and most clearly defined arcs. Loblolly pine, in contrast, has an indefinite diagram which shows only faint evidence of preferred orientation.

The intensity curves (Fig. 2), plotted from microdensitometer readings, show more convincingly that a large proportion of the crystallites are in almost perfect orientation with the remaining crystallites in another group of preferred orientation. These curves have all been reduced to the same scale for purposes of illustration. The inner curve (1) is fir; where the first ordinate for the top crosses this curve is the location of 5° deviation from perfect on the abscissa. This same point for spruce is at an angle of 10.5°; for hemlock, 13.5°; for cedar, 19°; and for pine, 23°. This means that the same percentages of the cellulose crystallites in the five fibers are oriented to the degrees indicated from perfect orientation. The curves may be redrawn as in Fig. 3 from calculations of the percentage of crystallites over each 5° angular range. Only one quadrant or 90° is required [that is from 0 to 90° to the fiber axis (or from intensity values from the 12 to 3 o'clock positions)], but checks can be made by the use of values from 3 to 6, 6 to 9, and 9 to 12 o'clock positions, since these should be uniform. The intensities for each 5°, corrected for background fogging on the film, are added and the ratio of any given value to the total summation gives the percentage.

A later study made with the spring- and summerwood pulp fibers of Douglas fir showed that the fiber orientation of the summerwood fibers is vastly more perfect than is the case for the springwood fibers. This should be reflected in increased tensile strength, resistance to chemical action, and other similar properties. Experience shows that the summerwood pulps possess high tear and low bursting strength, whereas the springwood pulps are high in bursting strength and low in tear.

The application of these x ray data to the physical properties of pulps and paper has not as yet been

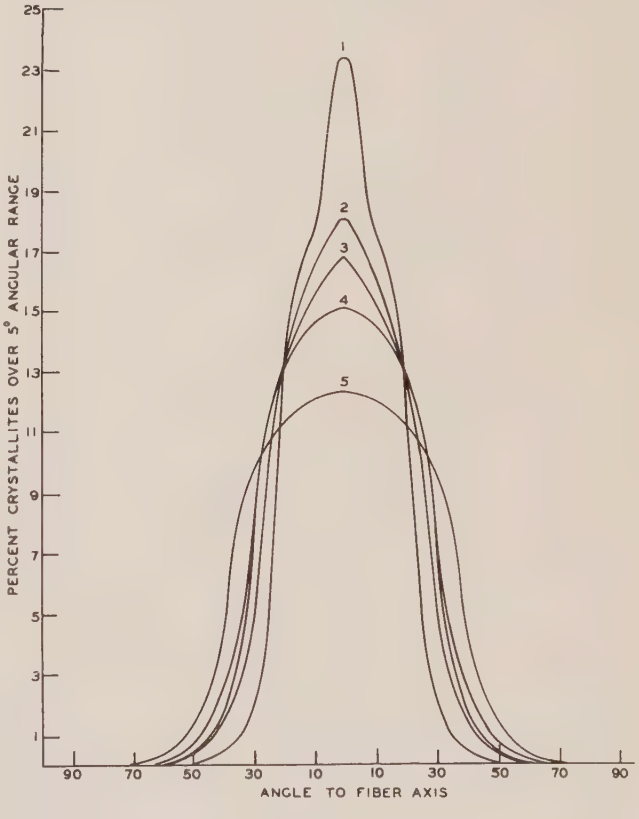


***Fig. 3. Preferred orientation in pulp fibers as indicated by the percentage of crystallites over 5° angular ranges as a function of the angle to the fiber axis***

[No. 1 (Douglas fir) is mostly highly oriented, and No. 5 (loblolly pine) is most nearly random.] (1) Douglas fir; (2) spruce; (3) hemlock; (4) cedar; and (5) loblolly pine.

worked out. Certainly a very large field of research is indicated and results of value in explaining the strength properties of pulps may be expected.

A large volume of work has been done relating the properties of natural textile fibers and their x ray structures. As orientation increases, the luster increases and the depth of dyeing decreases. The tensile strength increases and elongation decreases. In ramie, the cellulose crystallites have an orientation almost parallel to the fiber axis. Ramie fibers have the greatest tensile strength of all natural fibers but are relatively brittle and weave into a harsh fabric. Alignment makes for strength and rigidity in the fiber direction but brittleness across the fiber. In cotton, the crystallites are inclined at an angle to the fiber axis; cotton does not have the tensile strength of ramie but is resilient and weaves into a softer fabric. The crystalline part of the fiber is responsible for tenacity, resistance toward bending, and high modulus of elasticity. Amorphous parts cause softness and are concerned with swelling.

Thus, it is logical to interpret the Douglas fir pattern as indicative of higher fiber tensile strength, lower pliability and elasticity, and lower swelling and felting properties. In a general way, these are the characteristics of Douglas fir. The pine picture, on the other hand, suggests a pulp fiber of the opposite characteristics.

Whatever the application and significance, here is an outstanding difference between Douglas fir and the other pulp fibers.

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# The Characteristics of Unbleached Kraft Pulps from Western Hemlock, Douglas Fir, Western Red Cedar, Loblolly Pine, and Black Spruce

## *VII. Comparison of Springwood and Summerwood Fibers of Douglas Fir**

W. F. HOLZER and HARRY F. LEWIS

**Springwood and summerwood of Douglas fir were separated by hand methods and cooked separately by the kraft process. Springwood pulp is characterized by a high bursting strength, low tear, and considerable cutting of the fiber in beating. Summerwood pulp is characterized by a low bursting strength, high tear, and only mild fibrillation in beating. Specific surface of springwood pulp is initially quite high, changes very little on beating during most of the cycle, and then increases rapidly toward the end. Summerwood pulp has a low specific surface initially which increases regularly with beating and rapidly at the end of the cycle to values higher than those of springwood.**

A PROMINENT visual and structural characteristic of Douglas fir (*Pseudotsuga taxifolia*) wood is the abrupt change from springwood to summerwood in the annual rings. In addition the summerwood region is unusually hard for a coniferous tree and is made up of fibers having thick walls with almost no lumen; whereas the springwood region is much softer and fibers from it are large in diameter with much thinner walls and large lumens.

In a study at the U. S. Forest Products Laboratory in 1938 (*1*) the springwood and summerwood of southern pine were separated by hand, small amounts pulped separately, and handsheets made from the unbeaten pulps. The springwood sheets were hard and dense with a smooth surface while those from summerwood were soft, bulky, and had a rough, wooly surface. The nature of the rings of Douglas fir indicated that such an experiment on this species would be desirable in an extensive study of the physical and chemical properties of the wood and pulps, and would probably yield information on the relation of wood properties to the pulp properties.

### EXPERIMENTAL

Chips of separated springwood and summerwood were prepared by hand. First, the blocks were sawed into $^3/_4$-inch cards at right angles to the grain, and these cards were chipped with a chisel, alternately cutting off a springwood and a summerwood portion.

This hand separation was facilitated by selection of a wood sample that had annual rings averaging $^1/_4$ to $^3/_8$ inches in width, divided about equally between springwood and summerwood. About 4 pounds dry weight of each sample were prepared.

Kraft pulps were prepared in a stainless-steel autoclave with a capacity of about 4 liters which was heated by laboratory gas burners. The autoclave was protected from the direct flame and heat was distributed up the sides by a steel baffle at the bottom, and a jacket, asbestos insulated externally, which left an air space of about an inch around the autoclave. A valve for relief, a pressure gage, and a thermometer well constituted its fittings.

The chips, having become air-dried in preparation, were soaked in water over night before cooking. The chips were charged into the autoclave, and chemical amounting to 17 to 19% of active $Na_2O$ on the basis

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* This work was carried out by the members of the staff of the Central Research and Technical Department, Crown Zellerbach Corporation, Camas, Washington, and of The Institute of Paper Chemistry, Appleton, Wisconsin.

of dry wood was added together with sufficient water to cover the chips.

The autoclave was heated with gas burners to 175°C. in 90 minutes, and held at temperature for 45 minutes. Air was relieved at temperatures below boiling and finally swept out by steam.

At the end of the cook the pressure was relieved rapidly to atmosphere, and the pulp was washed, disintegrated, and screened. Pulp and screenings yields were determined and permanganate numbers and beater tests run on each pulp. Tests were run according to standard TAPPI procedures T 214 m-42, T 200 m-45, T 205 m-47, T 227 m-46, and T 220 m-46, respectively.

At each beating interval a sample was taken for specific surface measurements at The Institute of Paper Chemistry. These tests were run according to Institute Method 419 modified as described in the Research Bulletin (Institute of Paper Chemistry) (*2*).

## DISCUSSION OF RESULTS

Density tests run on the chips gave expected but rather startling results (see Table I). Summerwood with a density of 39.1 pounds per cubic foot was over twice as heavy as springwood with a density of only 18.6 pounds per cubic foot.

Pulp yields were low and erratic due to high screenings from the dry chips, and poor circulating conditions in the autoclave. No significant differences are visible in the data.

In the autoclave it has been found desirable to cook with rather high chemical charge because of the high dilution necessary to cover the chips with liquid. It will be noted that the springwood on a similar time temperature cycle, and an even higher chemical charge tended to cook considerably slower than the summerwood as evidenced by a permanganate number of nearly 35 for the former as against one of only 25 for the latter.

Fractionation in a Bauer-McNett Classifier showed that the summerwood had an appreciably higher percentage of very long fibers, but the two pulps were almost identical in the short fractions.

It should be noted that a microscopic check of the pulps indicated an effectiveness of about 80% in separation of springwood and summerwood fibers.

Handsheets made from the two pulps were decidedly different in properties (Table II, Fig. 1). Springwood,

**Table I. Cooking and Pulp Data**

| | *Springwood* | | | *Summerwood* | | | *Whole Wood* | |
|---|---|---|---|---|---|---|---|---|
| Wood density, lb./cu. ft. | 18.6 | | | 39.1 | | | 27.2 | |
| Cook No. | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 |
| Active $Na_2O$ on wood, % | 17 | 19 | 19 | 17 | 17 | 17 | 18 | 18 |
| Time to 175°C., min. | 90 | 90 | 80 | 90 | 105 | 90 | 95 | 90 |
| Time at 175°C., min. | 45 | 45 | 50 | 45 | 40 | 45 | 45 | 45 |
| Permanganate No. | 36.5 | 34.8 | 33.0 | 25.5 | 26.1 | 24.6 | 26.7 | 28.8 |
| Screened pulp, % | 35.9 | 36.9 | 36.9 | 42.2 | 36.4 | 33.4 | 37.5 | 41.1 |
| Screenings, % | 14.5 | 12.3 | 7.7 | 6.1 | 4.5 | 11.3 | 11.0 | 6.8 |
| Fractionation: | | | | | | | | |
| Retained on 14 mesh | 30.8 | | | 44.2 | | | 36.6 | |
| Retained on 20 mesh | 39.2 | | | 29.2 | | | 34.7 | |
| Retained on 35 mesh | 20.5 | | | 17.8 | | | 21.1 | |
| Retained on 150 mesh | 6.7 | | | 6.7 | | | 6.4 | |
| Passed 150 mesh | 2.8 | | | 2.1 | | | 1.2 | |

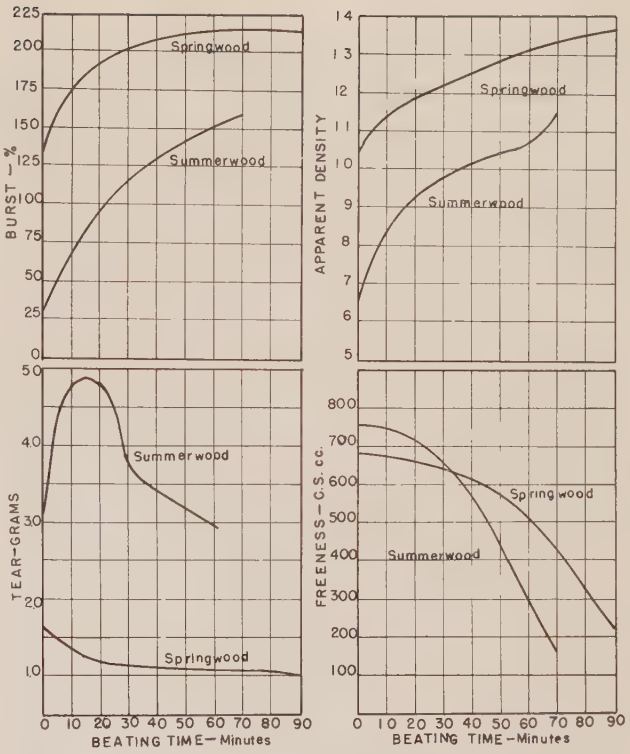


***Fig. 1. Physical tests of springwood and summerwood pulps***

even unbeaten, gave a hard, tinny sheet and became quite translucent on beating. Summerwood, unbeaten, was very soft, bulky, and had a very rough surface. After beating, it resembled the unbeaten springwood in texture.

Tests were in the ranges that would be predicted from the appearance of the sheets. Springwood had a very high-initial burst, and on beating developed this test to excellent levels, but its tear was comparable to sulphite pulps. Burst of summerwood started low and rose slowly to a much lower maximum, but the tear was exceptionally high. Apparent density (basis weight divided by the caliper in mils) followed the burst curves. For springwood it started high and rose to still higher levels. For summerwood, it started very low, became equal to the initial springwood sheet only after 40 minutes beating, and required 70 minutes beating to equal the 10-minute springwood sheet.

At the end of the beating treatment, the thick-walled summerwood fibers exhibited almost no cutting and only a mild degree of fibrillation, even though the stock had become so slow that it was difficult to make a sheet. Springwood, on the other hand, was severely cut and extensively fibrillated. These observations are in close agreement with the tearing strengths of the respective pulps.

Specific surface measurements made on samples of these pulps taken at various times in the beating cycle are fairly regular and as consistent as experience with this procedure would lead one to expect (Table II, Fig. 2).

On the springwood the specific surface had an initial value of 15,050 square centimeters per gram, rose slightly on 10 minutes beating, then remained substantially constant for 50 minutes, and finally rose sharply to 23,360 square centimeters per gram at the end of 90 minutes beating. The specific surface of

summerwood pulp started much lower at 10,045 square centimeters per gram, rose regularly with beating until it equaled that of springwood pulp at between 50 and 60 minutes beating, and then rose rapidly to reach 24,530 square centimeters per gram after 70 minutes beating.

When specific surface is compared to freeness, it will be seen that springwood fiber is consistently freer at a given surface than summerwood fiber. This seems in logical agreement with the observed tendency of springwood fiber to cut on beating while summerwood fiber tends to develop a very fine fibrillation.

Both burst and apparent density when plotted against specific surfaces give similar curves. The initial tests of springwood fiber are almost identical to the 40-minute sample of summerwood fiber. The springwood then maintains a much higher value for a given surface than the summerwood, and at freenesses in the range of 300 to 400 ml., both show a tendency for the fiber to increase rapidly in area without corresponding change in other tests. This seems in agreement with the usual bursting strength development curve in which the general breakdown of the fiber structure at about this freeness reduces ultimate fiber strength faster than fiber-to-fiber bond can be increased, and the curve reaches or approaches a maximum.

An x ray diffraction study was made on the Douglas fir springwood and summerwood through the cooperation of George L. Clark of the University of Illinois. Using a special technique in which small bundles of parallel fibers were examined, it was found that the summerwood fibers exhibited an unusually high degree of preferred orientation among the crystallites, whereas springwood fibers were found to be much more amorphous.

**Table II. Physical Pulp Tests**

| *Beating time, min.* | *Burst, %* | *Tear, g.* | *Apparent density* | *Freeness C.S., ml.* | *Specific surface, sq. cm./g.* |
|---|---|---|---|---|---|
| | | Springwood | | | |
| 0 | 127 | 1.60 | 10.2 | 685 | 15,050 |
| 10 | 174 | 1.30 | 11.4 | 675 | 16,360 |
| 20 | 191 | 1.11 | 11.8 | 660 | 16,320 |
| 30 | 197 | 1.13 | 12.2 | 650 | 16,780 |
| 40 | 206 | 1.08 | 12.5 | 610 | 15,850 |
| 50 | 208 | 1.06 | 12.9 | 580 | ..... |
| 60 | 208 | 0.98 | 12.8 | 515 | 16,960 |
| 70 | 214 | 1.05 | 12.6 | 425 | ..... |
| 80 | 222 | 1.02 | 13.6 | 315 | 20,130 |
| 90 | 216 | 0.94 | 13.7 | 215 | 23,360 |
| | | Summerwood | | | |
| 0 | 26 | 2.70 | 6.4 | 745 | 10,015 |
| 10 | 66 | 4.79 | 8.3 | 745 | ..... |
| 20 | 94 | 4.79 | 9.4 | 715 | 13,045 |
| 30 | 114 | 3.70 | 9.8 | 665 | ..... |
| 40 | 128 | 3.44 | 10.2 | 575 | 14,990 |
| 50 | 139 | 3.23 | 10.5 | 440 | 16,665 |
| 60 | 152 | 2.90 | 10.6 | 290 | 17,845 |
| 70 | 160 | 2.63 | 11.4 | 145 | 24,530 |
| | | Whole Wood | | | |
| 0 | 58 | 3.59 | 7.3 | 720 | ..... |
| 10 | 101 | 4.33 | 9.0 | 715 | ..... |
| 20 | 129 | 2.87 | 9.7 | 700 | ..... |
| 30 | 143 | 2.59 | 9.9 | 670 | ..... |
| 40 | 157 | 2.50 | 10.5 | 625 | ..... |
| 50 | 162 | 2.25 | 10.7 | 575 | ..... |
| 60 | 167 | 2.23 | 10.9 | 485 | ..... |
| 70 | 172 | 2.01 | 11.2 | 375 | ..... |
| 80 | 183 | 1.94 | 11.3 | 280 | ..... |

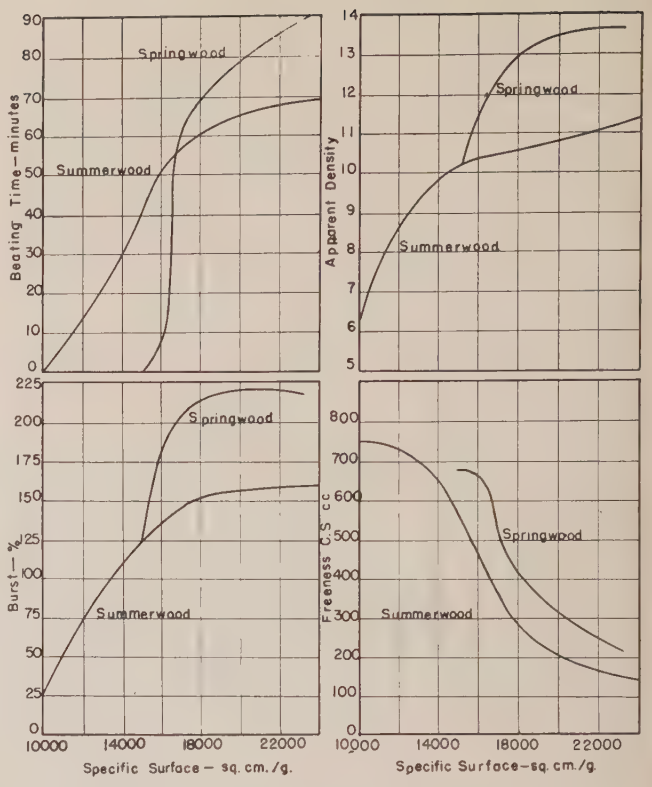


***Fig. 2. Relation of specific surface to physical properties***

This result is in excellent agreement with the characteristic behavior of the two fibers as reported here. A greater degree of molecular orientation would logically cause:

1. Stiff fibers, resulting in a bulky sheet with rough surface.
2. Reduced surface activity, resulting in weaker fiber-to-fiber bonds and lower burst.
3. Greater resistance to beating, as indicated by the low amount of cutting and only fine fibrillation of the summerwood fibers.

All these were the characteristic behavior of the summerwood fibers.

There has been almost no application of x ray diffraction studies of fibers in the field of paper, other than by analogy. While the results on this species may be unusual, they are certainly suggestive of a new field of research.

## CONCLUSIONS

The physical separation of Douglas fir springwood and summerwood fibers, even in 80% purity indicates that pulps from this species at least, are a mixture of two very different types of fiber. This type of study has been very little used probably because of the tedious techniques involved in the separation. The results are, however, indicative of a method that might well explain some of the seeming anomalies in the behavior of various samples of wood even in the same species.

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# PERSONAL MENTION

***Items About New and Old Tappimen***

## *New TAPPI Members*

*C. Gerald Albert*, Technical Director, Edgar Brothers Co., McIntyre, Ga., a 1935 graduate of Johns Hopkins University with a Ph.D. degree.

*Arthur Aronoff*, Student, New York State College of Forestry, Syracuse, N. Y.

*Marshall L. Barker*, Consulting Engineer, Beckett Paper Co., Hamilton, Ohio, a 1921 graduate of Rensselaer Polytechnic Institute with an M.E. degree.

*Larry D. Brice*, Engineer's Assistant, Union Bag & Paper Corp., Savannah, Ga. Attended Georgia School of Technology.

*Willard E. Carlson*, Student, The Institute of Paper Chemistry, Appleton, Wis., a 1947 graduate of Iowa State College with an M.S. degree from Lawrence College in 1949.

*Pauline C. Y. Chu*, Student, University of Maine, Orono, Me., a 1944 graduate of Ginling College, China.

*Sydney A. Collicutt*, Superintendent of Technical Services, Powell River Co. Ltd., Powell River, B. C., Canada, a 1939 graduate of British Columbia College.

*John R. Crawford*, Research Dept., Pabst Brewing Co., Milwaukee, Wis., a 1941 graduate of Tufts College.

*Gustavo Delfino*, Technical Director, C. A. Fabrica de Papel de Maracay, Caracas, Venezuela.

*LeRoy S. Dieffenbach*, Chemical Engineer, Robert Gair Co., Uncasville, Conn., a 1949 graduate of the University of Maine.

*John F. DuBeau*, Chief Chemist, Somerville Ltd., Crumlin, Ont., Canada, a 1938 graduate of University of Toronto with an M.A. degree in 1939.

*William H. Funk*, Development Engineer, Lukens Steel Co., Coatesville, Pa., a 1949 graduate of Drexel Institute of Technology.

*Raymond A. Greene*, Sales Representative, Monsanto Chemical Co., Merrimac Div., Everett, Mass., a 1947 graduate of Virginia Polytechnic Institute with an M.S. degree.

*Lana K. Hammond*, Serials Librarian, Denver Public Library, Denver, Col.

*John D. Hankey*, Technical Assistant, The Institute of Paper Chemistry, Appleton, Wis., a 1948 graduate of New York State College of Forestry.

*C. Raymond Humphries*, Research Chemist, Minnesota Mining & Manufacturing Co., St. Paul, Minn., a 1940 graduate of Pennsylvania State College.

*Irving H. Isenberg*, Research Associate, The Institute of Paper Chemistry, Appleton, Wis., a 1931 graduate of New York State College of Forestry, with a Ph.D. degree from the University of California in 1936.

*James H. Kirk*, Section Leader, Sinclair Refining Co., Harvey, Ill., a 1929 graduate of Pennsylvania State College.

*Louis O'D. Lee*, Consultant and Investor, New York, N. Y. Attended the University of Virginia.

*Lars E. Leje*, Assistant to President, Leje & Thurne A/B, Stockholm, Sweden.

*Gerald L. MacKinnon*, Supervisor of Raw Materials, Rogers Corp., Goodyear, Conn., a 1945 graduate of Massachusetts Institute of Technology.

*John M. Newton*, Director, Technical Sales Service, Clinton Foods, Inc., Clinton, Iowa, a 1941 graduate of Iowa State College.

*William E. Parfitt*, Sales Manager, Blaw-Knox Construction Co., Pittsburgh, Pa. Attended Carnegie Institute of Technology.

*Charles L. Peckham*, Technical Assistant, The Institute of Paper Chemistry, Appleton, Wis., a 1947 graduate of New York State College of Forestry.

*Donald V. Redfern*, Technical Director, American-Marietta Co., Seattle, Wash., a 1939 graduate of University of Washington.

*Carlos G. Robles*, President, Garcia Robles, S.A., Mexico City, Mexico, a 1927 graduate of the University of London.

*Orville K. Schmied*, Technical Director and Sales, Cochran Foil Co., Louisville, Ky., a 1932 graduate of the University of Louisville, with an M.S. degree in 1933.

*Eric R. Smith*, Staff Engineer, Alvin H. Johnson & Co., Inc., New York, N. Y., a 1925 graduate of Oregon State College.

*Frank J. Spinar*, Forest Products Economist, United States Army, NRS, GHQ., SCAP, Tokyo, Japan. Attended University of Wisconsin and Central YMCA College.

*Edwin I. Stearns*, Sectional Director, Calco Chemical Div., American Cyanamid Co., Bound Brook, N. J., a 1945 graduate of Rutgers University with a Ph.D. degree.

*James D. Stephens*, Sales Engineer, The Babcock & Wilcox Co., New York, N. Y., a 1942 graduate of Syracuse University.

*Victor J. Sutton*, Executive Director, Bowaters Development & Research Ltd., London, England.

*W. Arthur Thomas*, Plant Engineer and Assistant Mill Manager, Chillicothe Paper Co., Chillicothe, Ohio, a 1931 graduate of Tri-State College.

*William M. Thompson*, Chemical Engineer, West Virginia Pulp & Paper Co., Luke, Md., a 1939 graduate of West Virginia University.

*Freeling M. Truesdale*, Technical Service, Nekoosa Edwards Paper Co., Port Edwards, Wis., a 1927 graduate of University of Michigan.

*Harold D. Wells, Jr.*, Vice-President, American Paper Machinery & Engineering Works, Inc., Glens Falls, N. Y., a 1942 graduate of Union College, Schenectady.

*Francis O. Whipple*, Student, Oregon State College, Corvallis, Ore., a 1948 graduate of Reed College.

*Juan P. Zamboni*, Director General, Papelera Rio Parana S.A., Buenos Aires, Argentina. Attended Engineers Industrial University, Buenos Aires.

*Harry Zimerl*, Structural Engineer, Frederick L. Smith Co., New York, N. Y., a 1930 graduate of Cooper Union Institute of Technology.

## *TAPPI Notes*

*Harold R. Alley* is now in Okinawa as a civilian Industrial Engineer with the Department of the Army to head the industrial development activities for Military Government, Ryukyus Command.

*Le Roy J. Bauer*, Sales Engineer for the Black Clawson Co., is now with B-C International, Victoria, London, S. W. 1, England.

*Cornelius Betten, Jr.*, of the Champion Paper & Fibre Co., has been transferred from Canton, N. C., to the Research and Development Dept. at Hamilton, Ohio.

*Robert E. Bundy* is now Vice-President in charge of Production, Fibreboard Products, Inc., San Francisco, Calif.

*William H. Byron*, formerly of the N. Y. State College of Forestry is now a management trainee with the Hammermill Paper Co., Erie, Pa.

*Eugene F. Donnelly, Jr.*, formerly of the West Virginia Pulp & Paper Co., is now Assistant to the Plant Manager, Flintkote Co., Little Ferry, N. J.

*Bernard F. Hale*, formerly of Shartle Bros. Machine Co., is now with Chas. T. Main, Inc., Boston, Mass.

*W. F. Hathaway* is now Assistant Manager of Paper Manufacture, Kalamazoo Vegetable Parchment Co., Parchment, Mich.

*Ching-Yuin Hsu*, formerly of the University of Maine is now in the Technical Dept., Taiwan Pulp & Paper Co., Taipah, Formosa, China.

*Harold G. Ingraham*, formerly Chief Engineer for the Bird Machine Co., for 18 years has joined the staff of Chas. T. Main, Inc., Boston, Mass., in their Pulp and Paper Division.

*Hugo Klein* is now Vice-President of RC Reproduction Products, Los Angeles, Calif.

*Francis J. McAndrews*, formerly of Sonoco Products Co., is now Assistant to the General Superintendent, George O. Jenkins Co., Bridgewater, Mass.

*James H. McClure*, formerly of the St. Regis Paper Co., is now Chief Industrial Engineer, Sherman Paper Products Co., Newton Upper Falls, Mass.

*Charles A. Morton, Jr.*, formerly of Doubleday & Co., is now Service Engineer for the West Virginia Pulp and Paper Co., 230 Park Ave., New York, N. Y.

*Richard J. Murtaugh* is now Technical Director of the Dunn Paper Co., Port Huron, Mich.

*Leo P. Newton*, formerly of the Rushmore Paper Co., is now Assistant Plant Superintendent, Dunn Paper Co., Port Huron, Mich.

*Roy D. Rivers*, formerly of Industria, Inc., São Paulo, Brazil, is now Superintendent, Longview Fibre Co., Longview, Wash.

*M. E. Sanford* is now General Production Manager, Fibreboard Products, Inc., San Francisco, Calif.

*Arthur J. Saunders*, formerly of Sidney Roofing & Paper Co., Ltd., is now a Consultant on Manufacturing and Engineering at 317 Pemberton Bldg., Victoria, B. C.

*Ellman R. Smith* is now Mill Manager, Union Mills Paper Mfg. Co., New Hope, Pa.

*William A. Strieker*, recently of the Univ. of Illinois is now with the Atlas Mineral Products Co., Mertztown, Pa.

*George H. Whiteside* is now Paper Mill Superintendent, Container Corp. of America, Fernandina, Fla.

*George L. Witham*, formerly of Ciba Co., Inc., is now Sales Engineer for Orr Felt & Blanket Co., Mobile, Ala.

*Curt A. Young*, formerly of the West Virginia Pulp & Paper Co., is now Manager of Engineering, Riegel Paper Corp., Milford, N. J.

* * *

*J. A. Cauwels* has succeeded P. Williamson as the official representative in TAPPI of the Federal Paper Board Co., Bogota, N. J.

*Franklin F. Frothingham* has succeeded Harold G. Ingraham as the official representative in TAPPI of the Bird Machine Co., S. Walpole, Mass.

*R. E. Carter* has succeeded C. J. Kaiser as the official representative in TAPPI of Reichhold Chemicals, Inc., Detroit, Mich.

## *Industry Notes*

The Esselen Research Corp., 857 Boylston St., Boston, Mass., is now the Esselen Research Division of the U. S. Testing Co., Inc.

* * *

Frederic C. Clark Associates, Consulting Engineers, are now located at 141 E. 44th St., New York 17, N. Y.

* * *

The Dorr Co., has removed its executive offices from New York City to Barry Place, Stamford, Conn.

* * *

Paper & Industrial Appliances, Inc., has elected the following officers: President—Audenried Whittemore; Vice President—L. G. Durant; Secretary—Gudrum Sheffler; and Treasurer—Mrs. M. S. Kehoe. J. B. Beveridge consultant for the company was elected with Messrs. Whittemore and Durant to the Board of Directors.

* * *

F. E. Winslow is now Manager of the Baltimore, Md., office of the General Electric Co., Apparatus Dept., Atlantic District, succeeding J. W. Hicklin, retired. Mr. Winslow was formerly connected with the Philadelphia office and is a registered professional engineer of Maryland and Pennsylvania.

* * *

Stowe-Woodward, Inc., Newton Upper Falls, Mass., recently applied a Stonite covering to a 59 by 60 by 220 dual press center roll for the Crossett Paper Mills, Crossett, Ark. This is the largest diameter roll ever covered. It will be used in the manufacture of kraft and specialties.

* * *

The American Cyanamid Co., 30 Rockefeller Plaza, New York 20, N. Y., has published a 64-page revised edition of "Products and Services of American Cyanamid Co., for Industry and Agriculture."

* * *

The Elevator Division, Westinghouse Electric Corp., Jersey City, N. J., has issued a 44-page booklet (B-4402) on "How to Select Freight Elevators." There are ten illustrated sections that explain the basic considerations involved when selecting freight elevators and their component parts.

* * *

The Macbeth Corp., manufacturing operations are now centered in its new plant at Newburgh, N. Y.

* * *

Dilts Machine Works, Fulton, N. Y., has issued an 8-page bulletin No. 1-DM on the Miami Lo-Type Waxer and Water Finish Attachment, together with the Koehler Unwinding Equipment. This system can be used for both wet and dry wax processing.

* * *

The Sinclair Co., manufacturers of paper mill wire cloth and dandy rolls has appointed E. L. Epperson as sales service representative at Hamilton, Ohio. Mr. Epperson was formerly service representative in New England. Fred C. Jones has been appointed sales representative in the Midwestern area. David Sinclair has been placed in charge of all manufacturing in the Holyoke factory. Richard Sinclair is now sales manager and assistant to Peter S. Sinclair, President of the firm.

* * *

Cowles Chemical Co., Cleveland, Ohio, has moved its research and development laboratory from the campus of Syracuse University to 105 S. Townsend St., Syracuse, N. Y. The research projects include development of heavy chemicals in the silicate field, detergents, bactericides, and the engineering of new processes and equipment for three plants located at Lockport and Skaneateles, N. Y., and Sewaren, N. J. C. W. MacMullen is Technical Director.

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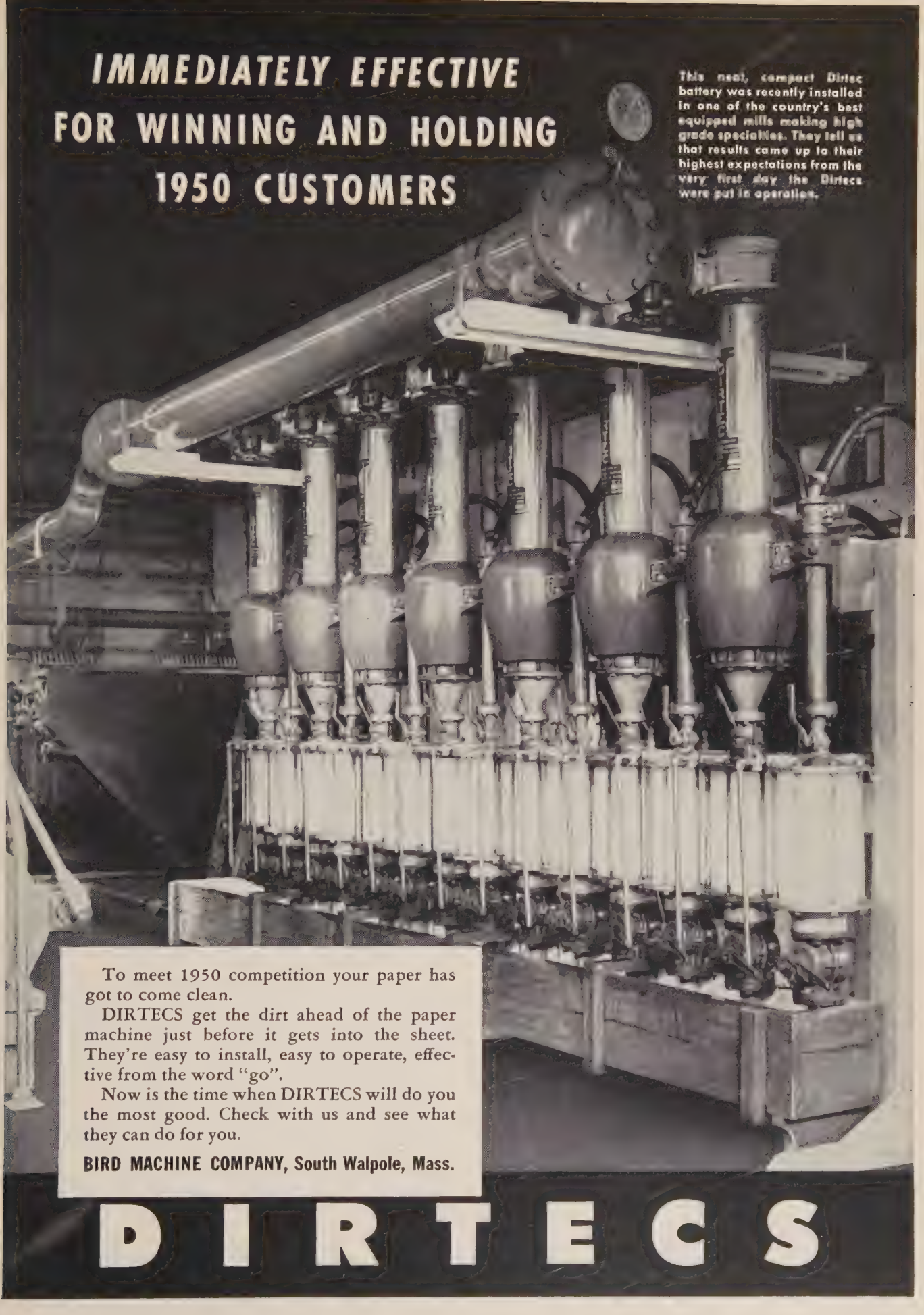
# 1950 CUSTOMERS



This neat, compact Dirtec battery was recently installed in one of the country's best equipped mills making high grade specialties. They tell us that results came up to their highest expectations from the very first day the Dirtecs were put in operation.

To meet 1950 competition your paper has got to come clean.

DIRTECS get the dirt ahead of the paper machine just before it gets into the sheet. They're easy to install, easy to operate, effective from the word "go".

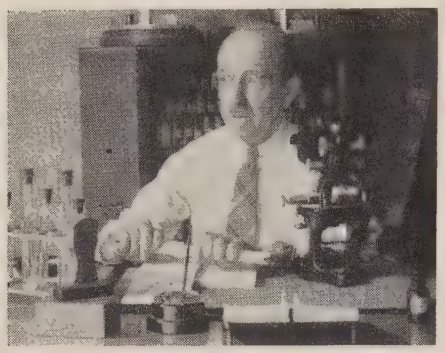
Now is the time when DIRTECS will do you the most good. Check with us and see what they can do for you.

**BIRD MACHINE COMPANY, South Walpole, Mass.**

# DIRTECS

# OBITUARIES

## John Henry Graff



John Henry Graff, 72, Research Associate, Emeritus of The Institute of Paper Chemistry, died suddenly from a heart ailment at his home in Appleton, Wis. on Dec. 30, 1949. He was born in Norway May 13, 1877, and came to the United States in June, 1904. His formal education included four years at the Military Engineering School in Oslo, Norway and one year at the Forestry School, Kongsberg, Norway.

His first five years in this country were spent working in two of the large department stores in Brooklyn. From there he went to Berlin, N. H. to do topographical drawing for the Brown Company After two years he was changed to the microscopic laboratory, and for twenty-two years headed up the work in microscopy. In January of 1934 he came to Appleton as a member of the staff of The Institute of Paper Chemistry to head the newly formed group in Fiber Microscopy which position he held until he was made "emeritus" in 1946. Since 1946 he has been carrying on research, filling special teaching assignments, and working on two books.

In his chosen field of fiber microscopy, he was an indefatigable worker, never satisfied with his own achievements, always seeking to improve. His laboratory technic was painstaking and meticulous, and he expected the same in his laboratory assistants and in his students. It may be said without exaggeration that no one man has contributed as much to the field of pulp and paper microscopy as has John Graff. The absence of any organized education and training makes his record even more impressive. In 1912 when he started his career in microscopy, it was generally sufficient for the fiber analyst to distinguish only between rag, groundwood, chemical pulp, bast and straw fibers. The situation rapidly became more complex with the introduction of new pulps; the analyst was asked to break the chemical pulps down into soda, sulphite, or kraft and to distinguish between bleached and unbleached and, in addition, to indicate the relative amounts of the various fibers. Then as new methods of bleaching and chemical refining were practiced, the situation became even more involved.

As the direct result of his efforts, John Graff was able to improve stains and technics and to develop the essential features of the method which presently serves for the identification and estimation of fibers cooked, bleached, or chemically refined by practically any method currently employed in the production of pulp and pulp derivatives.

He was not content to develop the methods, but he spread the gospel of their application with the zeal of an evangelist. The real reason he left the Brown Company to join the staff of the Institute lay in the opportunity it afforded him to teach his methods to young potential pulp and paper technologists. The extra work entailed in giving the TAPPI sponsored summer Fiber Symposium was cheerfully undertaken as a means of further spreading the same gospel. As a result, there are some three hundred technical men and women in the industry who have come under the influence of his enthusiasm and earnestness and who have gone out to use or apply the fruits of his research.

John Graff was a decided individualist. He was a good teacher and a good citizen. His small frame was surcharged with physical energy and nervous vitality. He was kindly, friendly, energetic—always planning a better way to do something whether it was in the laboratory or in his civic and personal interests.

The concentrated essence of his life time of service to the pulp and paper industry has been incorporated in some thirty technical papers, in three books, "Pulp and Paper Microscopy" (1936), "A Color Atlas for Fiber Identification" (1940), and "Fiber Analysis" (1942), in a number of patents and particularly in the manuscripts of two large works completed just before his death, one on "Reagents and Stains Used in Pulp, Paper, and Fiber Microscopy," the other "Pulp, Paper, and Fiber Microscopy." Shortly before his death, he started to outline a whole new program of research.

Because of his devotion to his field of research, he confined his outside interests to just a few organizations. One was Kiwanis, and a more loyal Kiwanian never lived. A second was in the field of photography, and during his years in Appleton he worked hard as a member of any organization of photographers in the area. His greatest extra-curricular enthusiasm was the Lakes States Section of the Technical Association of the Pulp and Paper Industry. This he served well over the years in whatever responsibility he was called to assume. As Vice-Chairman, Chairman, Chairman of its Program Committee he could always be relied upon. He looked upon his membership in the Technical Association itself as a serious matter. During his life he gave freely of his knowledge and his advice, as is shown by the number of his publications in the official organ of the Association and his contribution to the standard methods. His TAPPI summer Fiber Symposia were outstanding. It is fitting to point out that here was a man who gave much to the industry and whose name will never be forgotten. May there be more like him.

## Francis W. Johnson

Francis W. "Stub" Johnson, 58, plant manager and chief engineer for the Rhinelander Paper Co., Rhinelander, Wis., died, following an operation in St. Mary's Hospital in Rochester, Minn., on January 4. Mr. Johnson was born in Neenah, Wis., on Feb. 18, 1892. He studied mechanical engineering at the University of Minnesota. From 1913 to 1925 he was plant engineer and superintendent of construction and maintenance for the Kimberly-Clark Corp. From 1925–1934 he was chief engineer in charge of pulp and paper mill construction for C. R. Meyer & Sons Co., in Oshkosh, Wis. He joined the Rhinelander Paper Co. in 1934. He was a member of TAPPI. Surviving, besides his wife, are two sons Charles and William, a brother Donald W., a sister Mrs. S. B. Light and four grandchildren.

## Eugene A. Holland

Eugene A. Holland, President of the industrial advertising firm bearing his name, Philadelphia, Pa., was stricken with a fatal heart attack on January 10 at home. Mr. Holland was born in Philadelphia on June 11, 1900. From 1919–1921 he was asst. advertising manager for the Brown Instrument Co. From 1921–1928 he was Secretary of the R. E. Lovekin Corp.,

# for color in boxboard

## BISMARCK BROWN RX CONC. Dustless

BISMARCK BROWN RX CONC. DUSTLESS is tinctorially one of the strongest of basic colors. It is a standard requirement for producing browns and tans, and it acts as a dulling agent for the brighter Acid, Basic and Direct colors in both sized and unsized papers.

Although BISMARCK BROWN RX CONC. DUSTLESS does not work well on bleached fibers, its excellent solubility recommends it for both beater and calender coloring.

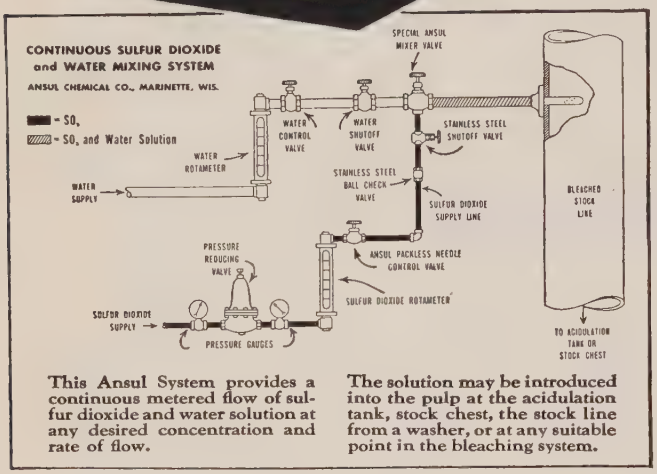
Specify BISMARCK BROWN RX CONC. DUSTLESS for such paper applications as kraft, poster, tissue, wrapping and boxboard.

AMERICAN Cyanamid COMPANY

CALCO CHEMICAL DIVISION,
Bound Brook, New Jersey

New York • Chicago • Boston • Philadelphia • Charlotte • Providence

FOR PERMANENT MAXIMUM
Brightness
in Bleached KRAFT Pulp
. . . INSTALL AN
ANSUL Constant $SO_2$
Concentration System . . . and get
Positive Control of Acidulation




This Ansul System provides a continuous metered flow of sulfur dioxide and water solution at any desired concentration and rate of flow.

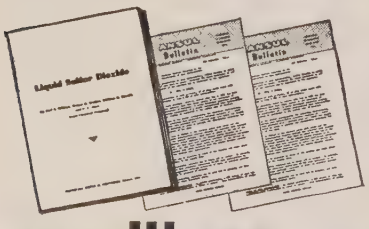
The solution may be introduced into the pulp at the acidulation tank, stock chest, the stock line from a washer, or at any suitable point in the bleaching system.

Ansul sulfur dioxide is an inexpensive acidifying agent . . . thoroughly efficient, and perfectly suited to the acidulation step in the preparation of bleached kraft pulp.

Ansul sulfur dioxide improves brightness and prevents color reversion because it:

1. Reduces colored ferric compounds to the soluble ferrous state so they may be easily washed from the pulp.
2. Bleaches and brightens the pulp by adjusting the pH to the acid side.
3. Acts as anti-chlor to remove residual chlorine and thus prevents formation of oxy-cellulose which would age to yellow compounds.
4. Permits dissolving of organic lime compounds so they may be removed by washing.

WRITE THE ANSUL TECHNICAL STAFF FOR FURTHER INFORMATION




Write today for these bulletins:
"A Method for Continually Preparing Sulfur Dioxide Solutions."
"Use of Sulfur Dioxide for Acidulation of Bleached Kraft Pulp."
Also, send for your copy of: "Liquid Sulfur Dioxide."

LIQUID Sulfur Dioxide ANSUL

ANSUL CHEMICAL COMPANY
INDUSTRIAL CHEMICALS DIVISION, MARINETTE, WISCONSIN
40 E. 42nd St., New York—Lincoln-Liberty Bldg., Broad & Chestnut St., Philadelphia 7, Pa.

and established his own business in 1928. His clients included the Asten-Hill Mfg. Co., Downingtown Mfg. Co., and the Sutherland Refiner Corp. Many in the pulp and paper trade who received the novel souvenirs from Asten-Hill will be interested to know about Mr. Holland's connection with these. He was a member of TAPPI and was unusually well informed concerning developments in the paper industry.

## E. H. Williams

E. H. "Bob" Williams, President of Williams-Gray Co., Chicago, Ill., died at Beverly Hills, Calif., on Dec. 17, 1949. Mr. Williams was born in Portland, Me., on July 27, 1870. Before the turn of the century he was agent for the sale of Meginticook paper machine felts and was a pulp salesman for Burgess Sulphite Fibre Co., Berlin, N. H. He established his own mill supply business in 1905. In 1908, with J. Frank Gray and D. C. Everest he formed the Williams-Gray Co., in which he was active until his death.

## Edouard Hery

Edouard Hery, owner and editor of Le Papier and of the machinery company in Paris, France, bearing his name, died at his home at Nevilly-sur-Seine on January 4. Mr. Hery, whose name was associated with the Thorsen-Hery beater, known in this country, was born at Angers, France, on Feb. 14, 1883. He attended the School of Arts and Metiers and in 1904 became engineer for S. A. des Papeteries Abadie, makers of cigarette paper. Later he was engineer for Papeteries Braunstein and Papeteries de la Seine. In 1910 he established his own engineering firm and represented such American firms in Europe as Bagley & Sewall, Farrel Foundry & Machine Co., Hamblet Machine Co., E. D. Jones & Sons, Wm. L. Barrell Co., Ryther & Pringle, Harman Machine Co., The Manhattan Rubber Co., and Sandusky Foundry & Machine Co. He was a member of TAPPI and a frequent visitor to the United States.

## Ernest John Kirkup

Ernest John Kirkup, Manager and Director of the Donside Paper Co. Ltd., Aberdeen, Scotland, died at his home in Aberdeen on Dec. 6, 1949. Mr. Kirkup was born at Woolaston, Gloucestershire, England, on Oct. 3, 1885. He started in the industry as a press boy on the paper machines of Thomas Owen & Co. Ltd., Cardiff, Wales, and served as machine foreman for Imperial Paper Co. Ltd., and Empire Paper Mills, Ltd., until in 1924 he became manager and director of the Donside Paper Co. Ltd.

# Bleach Liquor Prepared in 30 Minutes...

Preparing bleach liquor by introducing chlorine into excess milk of lime and waiting for it to settle is a time consuming process. In addition, it entails an expensive outlay for settling tanks and large storage facilities, the inconvenience of waiting and of sludge washing to say nothing of high labor costs. Nor can clear bleach liquor be used to buffer the bleaching operating beyond the solubility of lime.

These weaknesses in the preparation of bleach liquor were of concern to Hooker Technicians who realized that economies in pulp and paper production help Hooker as well as the industry. After a thorough investigation, Hooker Technicians, working with the pulp and paper industry, showed how a bleach liquor could be prepared so as to be used without settling and with resulting economies in equipment, labor and time. The key to this process is a low cost but efficient screen for the removal of all but the finest of the insoluble particles of lime before chlorination. Also important to the process is the agitation of the lime during chlorination and storage.

Coincidentally with the development of this process and its equipment, Hooker Technicians suggested the use of this same type of screen for introducing clean lime for "liming" in the chlorination stage.

- Hooker Bulletins 201 and 242 are devoted to a discussion of the production and use of unsettled bleach liquor and the equipment for this process. They are available when requested on your company letterhead.

HOOKER ELECTROCHEMICAL COMPANY
4704 BUFFALO AVENUE • NIAGARA FALLS, N. Y.
New York, N. Y. • Wilmington, Calif. • Tacoma, Wash.

HOOKER CHEMICALS

7-377 Caustic Soda Muriatic Acid Sodium Sulfide Paradichlorobenzene Chlorine Sodium Sulfhydrate

# LOCAL SECTION ACTIVITIES

***Reports of Meetings, Personnel and Events***

## *Inter-Society Color Council*

The Inter-Society Color Council, of which the Technical Association is a member will meet at the Statler Hotel, New York City, on March 8, 1950. A business session will be held in the morning. In the afternoon there will be a symposium on "Color As Used in Architecture, Design, and Decoration." The dinner speaker will be Ralph M. Evans who will give an illustrated lecture on "Seeing Light and Color." Members of TAPPI are welcome to attend all sessions. A. H. Croup of the Hammermill Paper Co., and Chairman of the TAPPI Optical Properties Committee is Vice Chairman of the Council.

## *Ohio*

The Ohio Section held its January dinner meeting at the Manchester Hotel in Middletown, Ohio, on Jan. 10, 1950, with 106 members and guests attending.

After the dinner, Chairman Arthur Thurn of the Champion Paper and Fibre Co. presented a guest, whom many were surprised to see and all were most happy to welcome, R. G. Macdonald, Secretary-Treasurer of TAPPI. Mr. Macdonald, spoke briefly about some of the activities of the Association and the publications in preparation. He also reminded the group of the coming 5th Annual Engineering Division Meeting in Cincinnati to be held on Oct. 2–5, 1950.

It was recalled, that Mr. Macdonald and the principal speaker of the evening Harry F. Lewis, Dean of Students at the Institute of Paper Chemistry, were both present at the organization meeting 15 years ago.

Dr. Lewis spoke on Chemical Byproducts of the Pulp Industry and developed the thought of what can be done and what has been done to reduce the hidden tax on each ton of cellulose produced, the tax represented by the other 50 per cent of the wood. These materials, in wood in addition to the cellulose, may be said to represent a tax which pinches pocket books in that they produce difficult waste utilization problems and are a loss of potential profits, if not recovered. The speaker stated that from two and one-half to three million tons of lignin sulphonate go to waste into the rivers each year in spent sulphite liquors, along with many tons of carbohydrates and other organic and inorganic constituents. These materials were shown to have many already known valuable uses as such, or as base materials for valuable derivatives.

Among the uses for spent sulphite liquor, which Dr. Lewis mentioned, was that to replace the oil treatment of dirt and gravel roads. Drenching the roads has been proved to be much better than the original spraying methods. Although this procedure has been proved to be effective, much more could be used if more widely accepted by those now using oil. Several of the uses given for the lignin sulphonate were its use for viscosity control in oil well drilling, its use to produce desirable properties in cement, its use as a substitute for tannin in the leather industry, and its use in the preparation of vanilin, the latter itself being valuable as well as a source for valuable chemical products. The speaker said, that the carbohydrates contained in such liquors are sufficient to fill 10 to 20 per cent of the country's requirements for alcohol. It was said that, although the mills in Sweden make little or no use of the lignin contained in their spent sulphite liquors, they do utilize a large portion of the liquors for the production of alcohol. Another development given was the production by one American mill of 9000 pounds per day of a high protein content yeast product for feed.

***Ohio Section Members at Dinner Meeting on January 10, 1950***

***L to R—Harry F. Lewis, R. G. Macdonald, and F. Le Roy Zellers at the Ohio Section Registration Table***

The kraft liquors, Dr. Lewis said, are valuable as fuel and as a part of an economic process, but the information developed for sulphite liquors can be utilized to develop uses for the kraft liquors also. Among some of the uses for kraft liquors mentioned, were the long list of fatty acid derivatives now being prepared. The saccharinic acids were mentioned as well as the possibility of converting the sulphur compounds to still other chemical products.

In conclusion, Dr. Lewis expressed a plea for more extensive active interest in the research and development field as well as in the chemical sales field.

The meeting was concluded with the showing of an educational and entertaining film in colors entitled "Portrait of an Industry." It was obtained through the courtesy of the Calco Chemical Division of the American Cyanamid Chemical Co.

The meeting held on February 9 was sponsored jointly by the Section and the Miami Valley Division of the Superintendents Association. Roland A. Pachard of the Holyoke Machine Co., Holyoke, Mass. presented a paper on "Paper Machine Power Requirements."

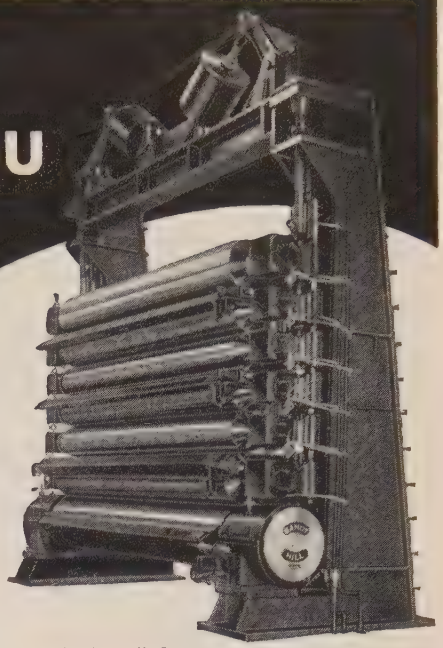
## *Lake States*

***December 13 Meeting:***

The Lake States Section met at the American Legion Club House, Appleton, Wis., on December 13. Robert A. Diehm of the Ward Paper Co., Merrill, Wis., talked on "The Elimination of the Human Element in the Determination of Printing Quality." Dr. Diehm said, in part:

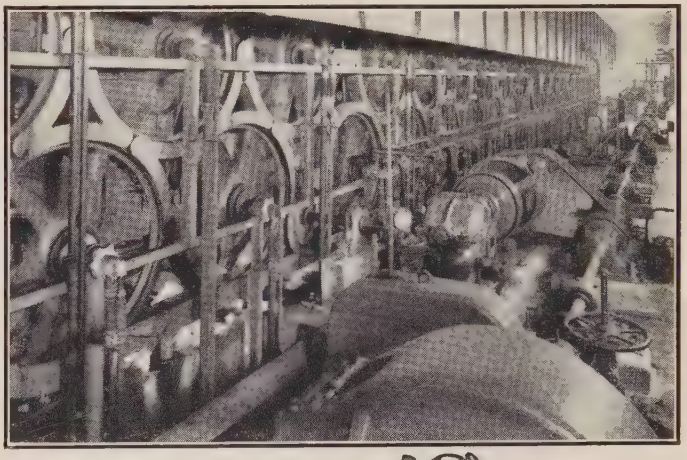
# *Sandy Hill* will work shoulder to shoulder with YOU

## in the interest of better paper and better paper making methods

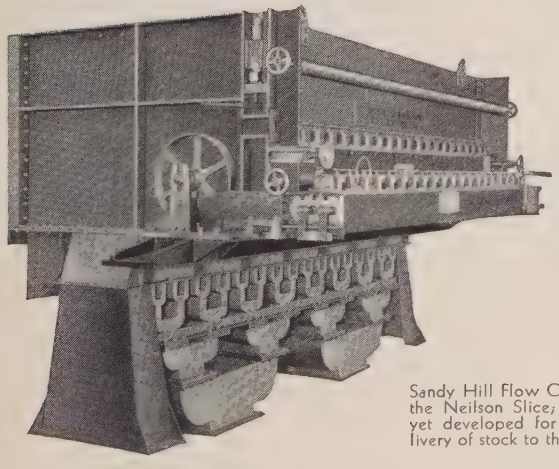


**Sandy Hill Open Side Calender Stack**
Pneumatic or hydraulic loading and lifting; continuous oiling; anti-friction or sleeve bearings, easy removal of rolls; sturdy fabricated framing.

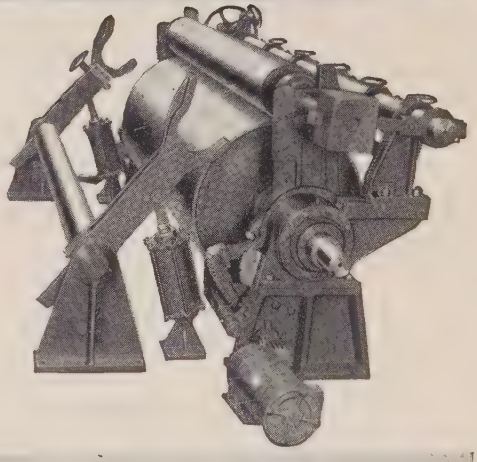
WITH advanced, pace-setting devices such as those shown here and many others, Sandy Hill has helped many mills step up production and make better paper faster and more economically. Perhaps there's a spot in your mill where Sandy Hill's engineering know-how can be of help . . . a kink to be straightened; a problem to be solved; a new idea to be developed. We're at your service . . . willingly . . . and, we've been told capably.



Typical installation of Sandy Hill Selective Drives, acknowledged leaders in power-packed performance, precision control and economy in space.



**Sandy Hill Pope Type Constant Speed Reel**
Motor control of reel transfer; anti-friction bearings throughout; pneumatic lift for full reel; shock absorbers for rough papers; design free of complications.



Sandy Hill Flow Control Unit, incorporating the Neilson Slice; the most precise method yet developed for uniformly controlled delivery of stock to the wire.

## Sandy Hill Iron & Brass Works

### Hudson Falls, N. Y.

*Machinists and Founders Specializing in Paper and Pulp Mill Machinery*

*Some Leading Items:*
Fourdrinier Paper Machines
Yankee Fourdrinier Machines
Cylinder Paper Machines
Feltless Wet Machines

Bertrams Flow Distributor
Kamyr Bleaching Equipment
Stock Pumps
Pulp Grinders
"Selective" and Corner Drives

Dandy Roll Drives
Quick Opening Gate Valves
Scofield Felt Conditioners
Cleveland Type Bearings
"Packer" Screens

Shaker Screens
Castings—Iron, Bronze, Aluminum
Neilson Slice
Nilsa Bag Machines
Nilsaton Printing Presses

# What do you look for in Paper?

"I looked at the quality of the paper finish after we put one Butterworth Calender Roll in the stack. I saw it was far better than previous finishes. It was smoother, of better quality, and our production was up with fewer delays. We've standardized on Butterworth Rolls since then, and we've had many additional hours of top flight calendering, increased production and less time lost."

Look into America's leading paper mills and you'll find Butterworth Calender Rolls producing outstanding finishes and giving long service. Butterworth Rolls are made of first quality materials and to exact specifications. That is why they can be depended upon to produce outstanding finishes during calendering operations.

With Butterworth Rolls in your calender, your own records will show you the economy of standardizing on these finer calender rolls with uniform surfaces, better finishes and better production.

Consult us about your calendering problems. We make calender rolls for all types of calendering in every size to your specifications. We can also refill your present rolls.

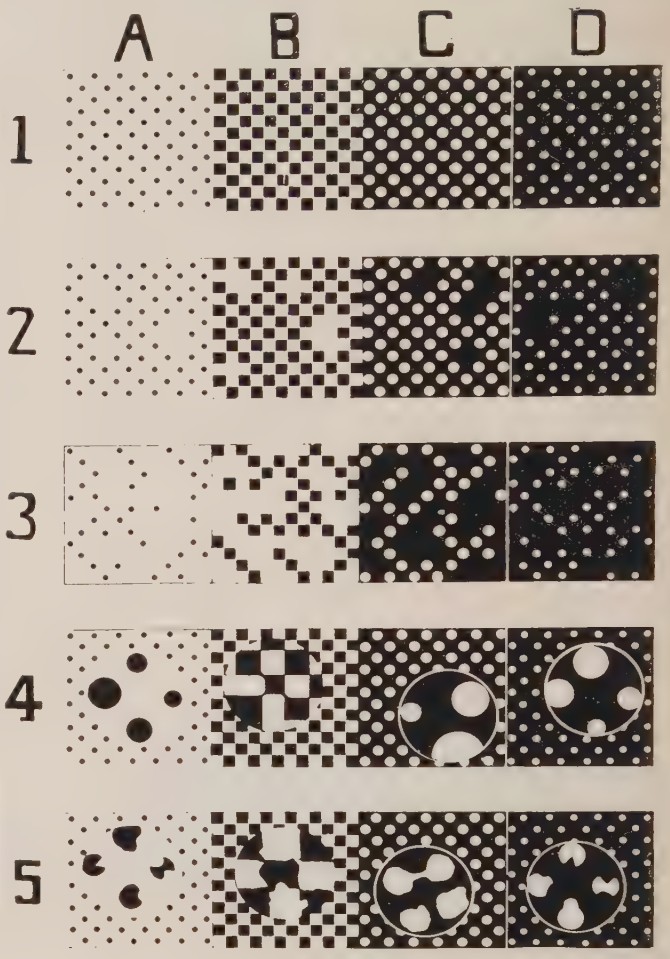
For full information, write or call H. W. Butterworth & Sons Company, Philadelphia 25, Penna.—Providence Division, Providence, R. I. : : Charlotte, N. C., 1211 Johnston Building In Canada—W. J. Westaway Company, Hamilton, Ontario

# Butterworth Calender Rolls

"Good jobs in every type of printing are obtained when sufficient amounts of ink can be applied to give tone gradations without altering the halftone screen pattern. Ink coverage and the screen patterns can be measured with the human element practically eliminated.

"A procedure to do this must be based upon a print job in which the ink and printing mechanism are held constant and the paper is made the variable. Any good laboratory proof press can be used. A heavy bodied, jet black ink should be used on a special printing plate. Ordinarily, the plate is inked as heavily as possible without disturbing the clarity of the high lights. Since register is not required, it is only necessary to maintain a constant temperature of the ink, paper, and press while making the prints.

"The plate is a typographical electrotype and is designed to show four tones in each of five different screens. The entire plate should cover an $8^1/_2$ by 11 in. sheet of paper to permit all the tests necessary to calculate the printing quality. The composition of the plate should follow the pattern shown in the accompanying figure:

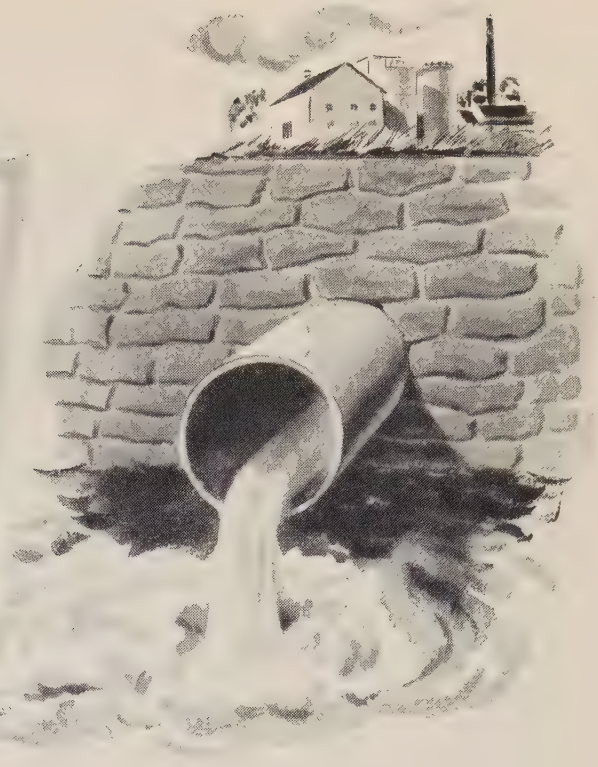



Row 1 is a 133 line screen
Row 2 is a 120 line screen
Row 3 is a 110 line screen
Row 4 is a 100 line screen
Row 5 is a 85 line screen

"This range of halftone screens covers the printing jobs from good newsprints at 85 lines to fine coated paper print jobs at 133 lines. Screens coarser than 85 lines are used for extremely rough work and screens finer than 133 lines are employed only in very sharp, delicate prints.

"The vertical rows have the following tones: Row A is 10%; Row B is 45%; Row C is 75%; and Row D is 90%. Before the halftone was used, a print or section of a print was either black or white. The gradations of tone made possible by the halftone screen are responsible for the exact reproduction of any subject. The 10% tone represents a high light in a print, meaning that only 10% of the paper surface is covered with ink. The 90% tone approaches a solid color and represents about the maximum color depth obtainable without closing the screen. The 45 and 75% tones represent two of the intermediate tone

# How to beat a legal "threat" on WASTE DISPOSAL



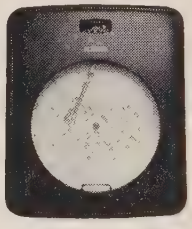
## . . . and cut fibre and chemical losses!

WITH STATE AFTER STATE now imposing legal restrictions on industrial waste disposal, you may be faced with this problem quite suddenly. Only by starting to gather complete data now can you be prepared for necessary action when that time comes. And the same data will reveal the extent of your fibre and chemical losses in plant waste. (The Foxboro waste-sampling system gives a continuous sample always in proportion to effluent rate—thereby assuring an accurate analysis.)

Foxboro has prepared a concise, informative bulletin on how to conduct a waste disposal survey. Based on specialized knowledge and long experience, this bulletin outlines the simple steps you can take to avoid hasty, last-minute plans. It shows how a careful, unhurried survey now can easily provide the facts on day-to-day quantities, concentrations, and constituents, so essential to efficient planning.

Send for a copy of "Waste Disposal Surveys". Write **The Foxboro Company, 78 Neponset Ave., Foxboro, Mass., U. S. A.**

**TYPICAL FOXBORO INSTRUMENTS THAT LEAD TO BETTER WASTE DISPOSAL**



The Model 40 . . . "The finest modern controller" . . . is the foundation of many outstanding Foxboro Waste Disposal Systems. It is used for the control of flow, pH, conductivity, temperature and other variables. Thousands are in use daily.

Foxboro Float and Cable Type Instruments are specifically designed for use on weirs and Parshall flumes. They read directly in *flow*.

FOXBORO
REG. U. S. PAT. OFF.

RECORDING · CONTROLLING · INDICATING
INSTRUMENTS

**holding power**
for short fibres

**PQ SILICATE**
REACTED IN THE BEATER
**TRAPS THEM IN**

PQ Silicate is the paper maker's friend. The precipitate which it forms with alum catches in the fibres and holds on to the little short ones so that they do not escape. Furthermore, this silicate size keeps more of the clay and other fillers right in the sheet. You profit in two ways:

1. **Improved quality — smoother, harder, and stronger sheets.**
2. **Increased output — as much as 30 lbs. per ton.**

May we send you further data and samples of the right PQ Silicate for your experiments? You're always welcome at PQ's Silicate Headquarters. For silicate advice, ask PQ.

**Philadelphia Quartz Co.,** 1141 Ledger Bldg., Phila. 6

**PQ SILICATES OF SODA**

**PQ WORKS:**
Anderson, Ind.
Baltimore, Md.
Chester, Pa.
Gardenville, N. Y.
Jeffersonville, Ind.
Kansas City, Kans.
Rahway, N. J.
St. Louis, Mo.
Utica, Ill.

gradations covered by the range of tonal values between the high lights and the full tones.

"From three to five prints should be made on each paper under investigation. The press operator should adjust the inking of the plate and the pressure of the press to give the best possible print job obtainable before making prints for analysis.

"The first step in analyzing the print consists in determining the ink coverage. This is done by measuring the brightness of all the 10 and 90% tones and obtaining the average for each. The ink coverage (IC) is calculated by:

$$IC = \frac{B\ 10 - B\ 90}{BP} \times 100$$

where

$B\ 10$ = the brightness of the 10% tone
$B\ 90$ = the brightness of the 90% tone
$BP$ = the brightness of the paper

"A perfect print has an ink coverage of 80% since the difference between the lightest and deepest tones is 80%. Reasonable variations in the inking of the plate do not influence the results because the increase in coverage between the 10 and the 90% tones is measured.

"The amount of ink laid on the surface of the paper tells only part of the story of printing. The patterns of the ink-covered areas are equally important. The finest screen which lays a perfect pattern suggests the type of printing for which a paper can be used. Alterations in the screen pattern may mean the difference between the sharp and fuzzy print. The next step in the analysis is the evaluation of the screen pattern in such a manner that concise measurements rather than human judgments are made.

"A print with a perfect screen pattern is sharp and has good print fidelity. Accurate observations on the screen pattern can be made with any hand lens that permits a clear perspective of the dot size and shape.

"In the first place, the black dots in the lighter tones and the white dots in the darker tones must all be present for good print fidelity. Only a few dots need to be absent before even the inexperienced eye can detect loss of fidelity in the print. It has been estimated that a print has little, if any, commercial value when more than 25% of the dots are missing.

"A value must be assigned to screen patterns on the basis of print fidelity with respect to the presence or absence of the halftone dots. When over 25% of the dots are missing, the tone is assigned a halftone dot fidelity of 0. If less than 25% of the dots are missing, the tone assumes a value of 1. When all the dots are present, the value is 2. All of the 20 tones are evaluated individually and the values totaled to obtain the rating for the halftone dot fidelity. A perfect print has a value of 40. Row 1 on the figure shows perfect printing in which each tone has a value of 2. Row 2 shows some dots missing to influence the printing enough to give each tone a value of 1. Row 3 shows enough dots missing to make the print worthless and those tones have dot fidelity of 0.

"The mere presence of the dots does not necessarily mean sharp printing. They must be of uniform size in a continuous tone and must not show distortion or broken contours in any tones. Broken and distorted dots are almost as detrimental to print fidelity as are missing dots. Here again it is necessary to make accurate observations and apply them in the calculation of the evaluation of the print.

"Using a hand lens, each of the 20 tones is examined for size of dot uniformity. When all the dots appear uniform, the tone is given a value of 2; when a few show variations in size, it has a value of 1; and when over 25% show variations in size, the value becomes 0. A perfect print is assigned a value of 40, being the sum of the values of all the tones. Such variations in dot sizes are shown in Row 4 in the figure.

"The tones are examined for undistorted and unbroken dot outlines with a hand lens. Row 5 in the figure shows some of the irregularities which reduce print fidelity. When such irregularities are absent, the tone has a value of 2; when a few are present, a value of 1; and when more than 25% of the dots are irregular, a value of 0. A perfect print again has a value of 40.

"The presence and the size and shape of the dots are all important in making a print of good fidelity. Since it would be exceedingly difficult to determine their relative importance, they are all given equal values of performance in this test. Therefore, the sum of all the fidelity ratings is 120 for a perfect print.

"These observations permit a new definition of printing quality based on ink coverage and print fidelity. *Printing quality is the per cent ink coverage obtainable in perfect print fidelity.* It is expressed numerically by:

$$PQ = \frac{IC \times PF}{96} \times 100$$

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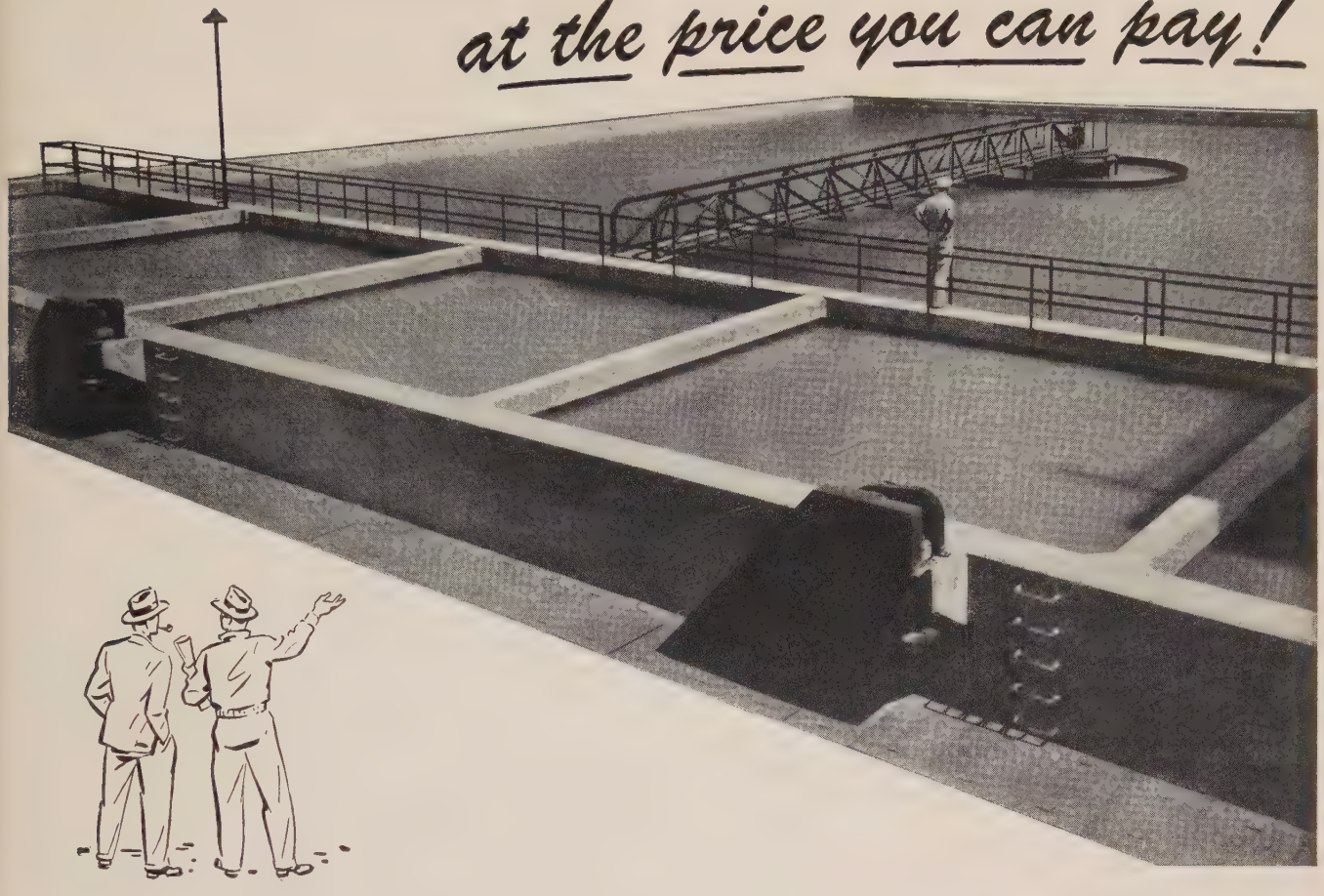

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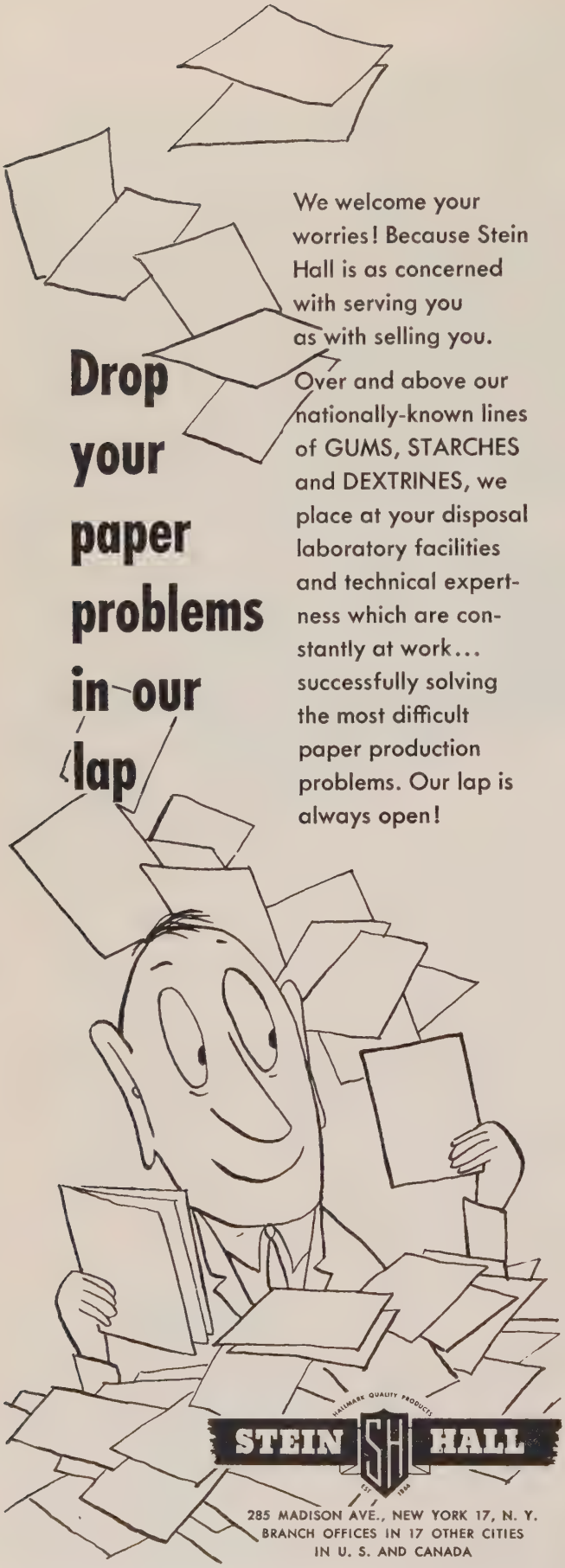
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Where

$PQ$ = Printing quality
$IC$ = Ink coverage
$PF$ = Printing fidelity
96 = Constant (per cent ink coverage in perfect fidelity in 100% printing quality, i.e., 80% of 120)

"This test can be carried out quite rapidly by laboratory technicians after they become thoroughly acquainted with it. The analysis of two printed samples is sufficient if the results are reasonably close.

"It has been mentioned previously that characteristics such as surface strength, caliper, density, porosity, sizing, formation, etc., specific to the paper for each print job must be met. Everyone connected with papermaking or printing can make measurements for these characteristics. If they are supplemented by the test described here, a much better understanding of the printing quality of the paper can be developed."

***January 10 Meeting:***

The Lake States Section met at the American Legion Club House, Appleton, Wis., on January 10. George R. Sears of the Institute of Paper Chemistry presided as chairman. Fred Benson of the Kimberly-Clark Corp., paid tribute to the memory of the late John Graff and commented on the great interest taken by Mr. Graff in the affairs of the Lake States Section as well as in the field of microscopy.

N. Malcove of the Northern Paper Mills, Green Bay, Wis., followed Mr. Benson with a talk on his recent trip to Europe. He was interested principally in the investigation of two things: (1) evaporation and burning of waste liquor and (2) making yeast from waste sulphite liquor. Sweden has done much research on these problems, and it was in this country that Mr. Malcove carried on the greater part of his inquiry.

The shortage of coal in 1940 in Sweden accelerated the investigation of the possibility of using waste sulphite liquor as a source of fuel. Many problems were encountered in evaporation, burning after concentration, and removing the fly-ash. Evaporators which were reasonably free from scale were developed by two companies. One employed the principle of interchanging the phases (Rosenblad), while the other submitted the waste sulphite liquor to heat treatment.

After eliminating the problem of evaporation, they found difficulties in burning waste sulphite liquor. It was attempted to burn liquor of 50% concentration and to burn dry powder. At least one plant has temporarily abandoned burning and is selling the liquor. Sweden prefers waste sulphite liquor to oil for use on her roads. The fly-ash, a result of burning, is removed in one of two ways, by wet scrubber or dynamic filter. Mr. Malcove stated that Sweden has been fairly successful with the evaporation problem, but there are difficulties to be solved in the burning of waste sulphite liquor.

The second thing Mr. Malcove investigated on his trip was the production of feed yeast from spent sulphite liquor. Sweden has ceased to produce this type of yeast and has dismantled one of her two yeast plants since the war. During the war Germany had at least five yeast plants which were all shut down by late summer in 1949. Two of these plants were back in operation in December of 1949 for the production of animal feed. Early production all over Europe was disposed of largely for human use. Mr. Malcove remarked that a market for yeast in the United States has yet to be developed.

JOHN D. HANKEY

## *Kalamazoo Valley*

The following is a résumé of the meetings of the Kalamazoo Valley Section held since June, 1949.

The first meeting was held at the Columbia Hotel, Kalamazoo, Mich., on October 6. Stephen I. Kukolich of the Lee Paper Co., Vicksburg, Mich., presided. The principal speaker was C. F. Hauck of the Hall Laboratories, Inc., Pittsburgh, Pa., who talked on "Water Management and the Paper In-

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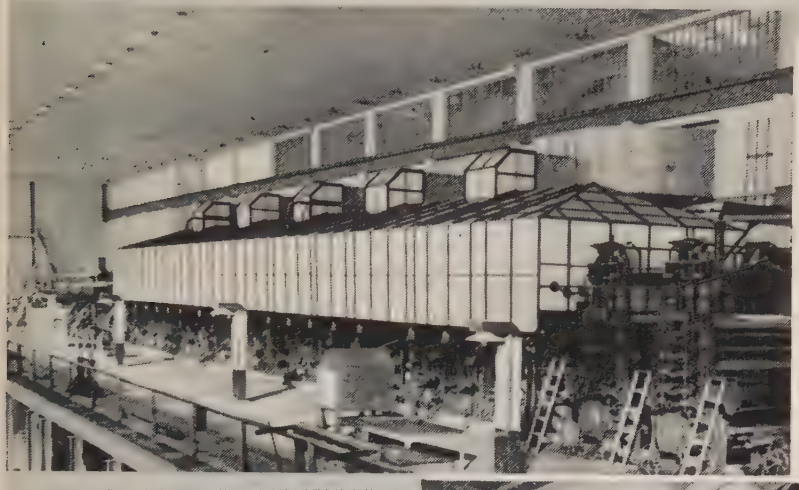

Ross Hood and Exhaust connections, showing ceiling outlets and Spot Cooling apparatus.. Machine is equipped with Briner Economizers, Ross-Grewin and Bottom Felt Supply Systems.

Inlet side of Ross Briner Economizer heating air by exhaust vapors from machine. This heated air is distributed through special ceiling outlets for roof heating.

Wherever in the world paper is made, ROSS Air Systems become an essential part of the paper-making process. They are practically standard mill equipment throughout the industry in the United States, Canada, Europe, Mexico, South America—and now Australia!

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In a country where fuel costs are necessarily high, the operation of ROSS Air Systems naturally proves highly profitable. It is significant that work is already underway on five other installations in Australia.

The Ross Briner Economizer apparatus on enclosed platform located over hood in back of No. 3 Machine.

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dustry." Dr. Hauck stated that "Development of a water balance showing water temperature, quality, and quantity characteristics at all points of water demand and discharge is the first step in the mill water management problem."

The meeting of November 3 was in charge of Ralph Atkins of the Lee Paper Co., who introduced Bruno E. Prevost of the Emerson Mfg. Co., Lawrence, Mass., as the speaker. Mr. Prevost described his "Jordan Plug Pressure Control System," illustrating the action with a working model. This talk was followed by a panel discussion in which W. F. Hathaway of the Kalamazoo Vegetable Parchment Co., J. J. Harrison of the Michigan Carton Co., and Ralph Atkins participated.

The meeting of December 1 was in charge of James J. Harrison of the Michigan Carton Co., Battle Creek, Mich. The principal speaker was H. A. Lips of E. I. du Pont de Nemours & Co., Wilmington, Del., who talked on "Shade and Quality Control in the Dyeing of Paper." This talk dealt with the fundamentals of beater dyeing of paper. Dr. Lips emphasized the need for close control of pulp furnishes and of the pH of the stock in the beater. He commented on the influences of the various fillers used on the dyes. Some dyes have an affinity for fillers as well as fibers. The end use of the colored paper should be known so that an economical formulation can be worked out. He pointed out that size, starch and their order of addition are factors influencing shade control in the dyeing of paper.

On January 12, the 15th Annual Get-together was held. This meeting which attracted an attendance of 425 was sponsored jointly by the Kalamazoo Valley Section and the Michigan Division of the Superintendents Association. Ralph A. Hayward of the Kalamazoo Vegetable Parchment Co., and a past national president of TAPPI was toastmaster. W. F. Hathaway of the same Company was general chairman. Several national officers of both organizations were introduced. R. G. Macdonald, Secretary-Treasurer of the Technical Association extended the greetings of that organization and Charles H. Reese of the Nekoosa-Edwards Paper Co. responded as President of the Superintendents Association. Robert B. Wolf, TAPPI Medallist was introduced and made a few remarks. The principal speaker was Jack T. Wilson, Chief Physicist of the Allis-Chalmers Mfg. Co., Milwaukee, Wis., who talked on "Atomic Power and Its Significance to America." Dr. Wilson outlined many of the peacetime possibilities of nuclear chemistry and the wide range of isotopes that will be developed.

On February 2, Fred Chappel of the Hercules Powder Co., was in charge of the program. The speaker was Richard W. Kingerly of DuPont who talked on "Biological and Pest Control for the Pulp and Paper Industry." In this talk Dr. Kingerly discussed the control of slime, fungi, bacteria, insects, and rodents. This talk was followed by a panel in which J. J. Harrison, Michigan Carton Co., G. F. Des Autels, Kalamazoo Vegetable Parchment Co., Harold O. Wiles, Sutherland Paper Co., H. R. Hall, Allied Paper Mills, and Ken Wyatt of DuPont participated.

## *Empire State*

### *Hudson River Valley Group*

The Hudson River Valley Group of the Empire State Section met at the Village Inn, South Glens Falls, N. Y., on January 19. John C. Dean, Manager of the Chemical Products Div., Lubricating Dept., Socony-Vacuum Oil Co., New York, N. Y., talked on "Waxes for Protective Coating."

### *Metropolitan Group*

On January 10 the Metropolitan Group of the Empire State Section met at the Fraunces Tavern in New York City. W. C. Nussbaum of Tracerlab, Inc., talked on "Uses of Radio Isotopes in Industry."

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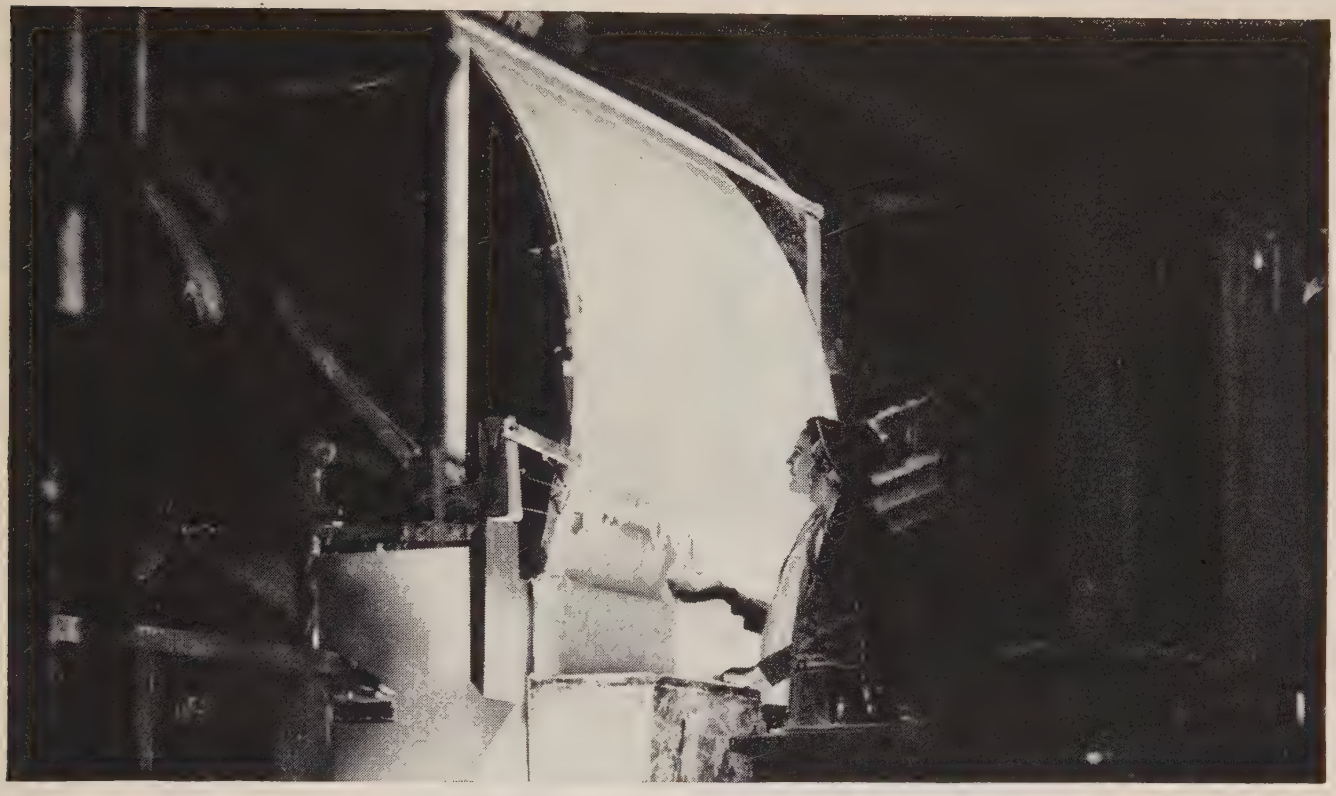

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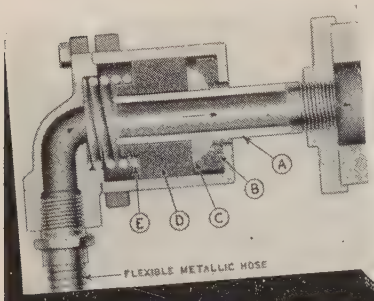


Nipple (A) is fastened to roll or drum. Carbon-graphite ring (B) effects seal against rotating collar (C). Guide (D) supports joint, is also of self-lubricating carbon-graphite. Spring (E) is for initial seating only; in operation joint is pressure-sealed.

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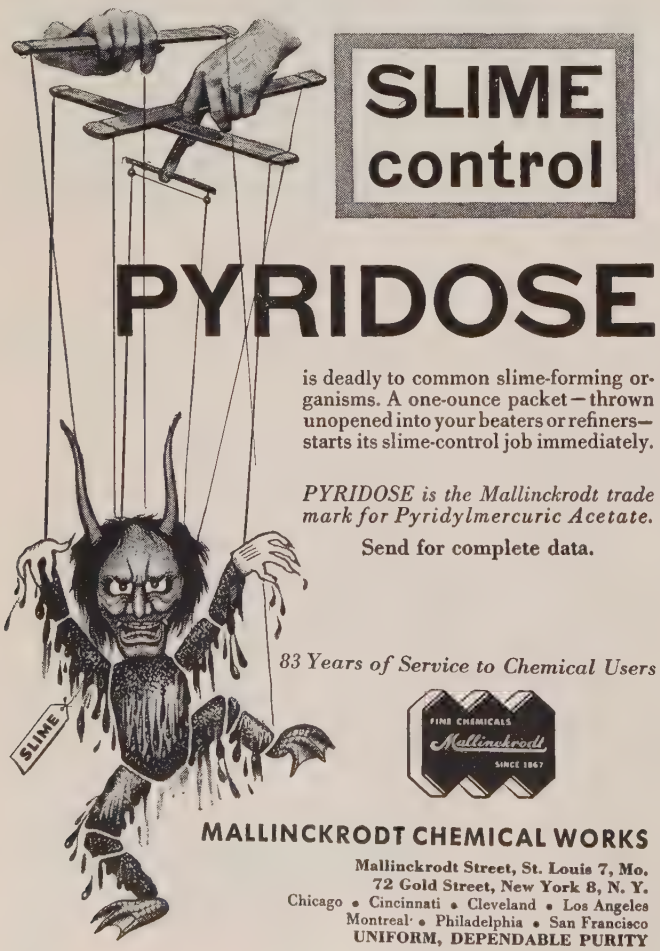


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*Northern New York Group*

The Northern New York Group of the Empire State Section met at the Hotel Woodruff in Watertown, N. Y. on December 8. John H. Heuer of the St. Regis Paper Co., presided as chairman. The principal speaker was Jess H. Davis, President of Clarkson College of Technology at Potsdam, N. Y., who talked on "Trends in Engineering Education." Dr. Davis' remarks follow:

"Engineers, both individually and as a group, are traditionally modest men who do not advertise their accomplishments. They have, however, had a most important part in the development of new products and processes which have brought improved living conditions to most of the world. It may be argued, perhaps with considerable justification, that the work of engineers has contributed in no small way to the reaction of many of the economic, political, and social problems which confront us today. There can be no argument, though, but that continued engineering and technological developments are essential to the progress and security of America.

"It is the function of the engineering colleges to formally educate men who, after training and experience, can join the engineering profession and insure further progress in the use of the materials and forces of nature for the benefit of mankind.

"Probably the first formal engineering education was given in France about the middle of the eighteenth century. It was of a most practical type of education in which the study of science was completely neglected and Empiricism was all-important. Soon after the French Revolution, science began to be emphasized and the character of French engineering changed rather abruptly from the practical to the theoretical. As often happens, the pendulum swung too far. The result was that French engineers were responsible for almost none of the applications of science to practical works during the nineteenth century.

"Little formal engineering education was offered in Britain during the nineteenth century; but that which was available could be described as of the 'applied science' type.

"West Point and Rensselaer, established around the beginning of the nineteenth century, were the first American engineering schools. They did not reflect the pattern of the French schools and, while they did attempt to teach engineering on somewhat of a scientific basis, the meager information relating theory to practice necessitated reliance on empiricism or experience for most instruction. From the middle of the nineteenth century until the beginning of the twentieth century, many schools were established. Rapid progress in relating theory to practice was being made during this period. There was, however, a strong vocational strain in most engineering education. There was little attempt made to teach the "why"; "how" was the important thing. This type of instruction continued to be the vogue in most engineering schools until about 1920, when wider use of electricity, the airplane, radio, and the automobile made it evident that there must be a much closer relationship between science and engineering if technological progress were to continue.

"Engineering research programs were instituted in many colleges to help bridge the gap between practice and theory. The trend of instruction was away from the empirical and toward instruction which would be of value for future uses rather than in past techniques. As might have been expected, however, there was much specialization during the 1920's. This trend in specialization has continued until very recent years. The emphasis, particularly in the past two or three years, has been more and more on fundamentals. In most engineering schools, the thinking is, "Let's educate a man to be an engineer first and a specialist in some branch of engineering later."

"Since the end of World War II, there has been a general reappraisal of engineering education. There has been no general agreement upon specific objectives or even upon how broad objectives can be best accomplished. There is a strong tendency on the part of most engineering schools to give an ever-increasing importance to science and fundamentals. There is also widespread recognition of the importance of developing creative ability and the maintenance and encouragement of intellectual curiosity.

"Engineering schools, in general, recognize the importance of the ability of the engineer to write and speak, and much attention is being given to this phase of engineering education. The importance of the human side of engineering has come into more general recognition, and the majority of engineering schools have developed a social humanistic program to give the student a better understanding of over-all human relations.

"A few schools have felt that their objectives in engineering education could be accomplished only by the establishment of a 5-year program, but the majority believe that four years is sufficient time in which to give the basic work that they consider necessary for the first degree.

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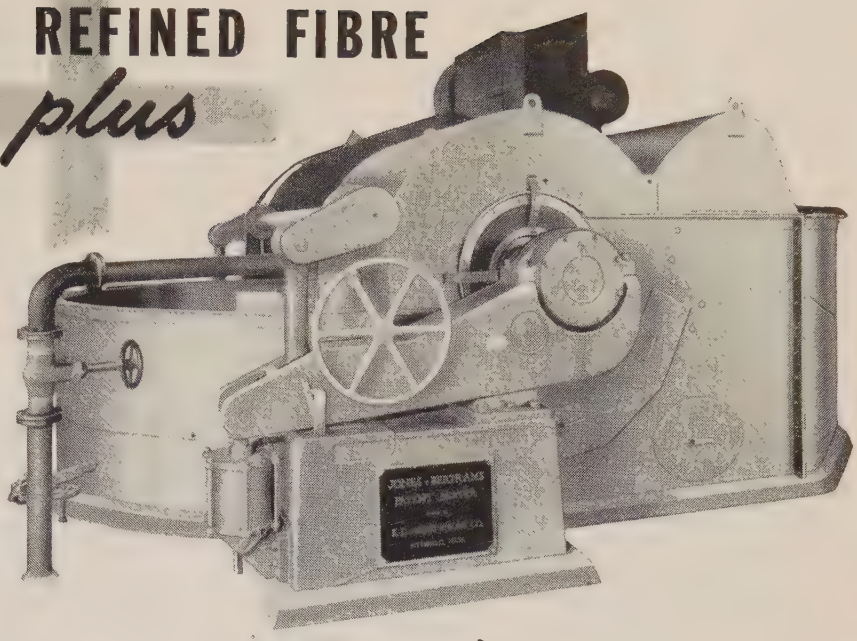

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"Cooperative education in engineering has many desirable features, but the practical details involved have made it difficult, if not impossible, for most schools to offer this type of program. There seems to be no distinct trend toward formal cooperative engineering education.

"Engineering is not a static profession. Engineering educators are aware of their responsibilities and they will continue to change engineering education so that it can best meet the changing needs of the profession."

***Meeting of January 12***

The Northern New York Group of the Empire State Section met at the Woodruff Hotel, Watertown, N. Y., on January 12. Harold Spector of the St. Regis Paper Co., presided as meeting chairman. The speaker of the evening Jerry Strasser, Manager of the Paper and Adhesives Dept. of Stein, Hall & Co., New York City, was introduced by H. A. Spencer of Knowlton Bros. Co., Watertown, N. Y. Mr. Strasser discussed "The Application of the Water Gels of Starch of the Manno-Galactan Origin." He pointed out that such additives as color, filler and size have varying effects on starch gums. He also indicated the necessity of using a complete designation to describe a gum since much confusion has arisen due to the wide and indiscriminate use of the terms gel and gum. The hydrophylic products were described and various applications of this quality were mentioned. He gave a brief sketch of their sources and processing, together with data in regard to their use.

Following his talk Mr. Strasser responded to questions. A few remarks follow:

*Question:* How do these manno-galactan gums act as beater additives?

*Answer:* We have done a lot of work where they have been a useful material. But in most cases where jordaning or heavy beating follows their addition to the furnish, most of the plus values that they may have are destroyed, although in certain lightweight papers they have been a very definite help in forming a tighter sheet.

*Question:* What effect has the manno-galactan gums on retention of fillers, etc.?

*Answer:* Their use reduces retention slightly. Fortunately, not seriously, however, A sheet having a normal 20% ash will be a 19+% with these gels added to the furnish.

*Question:* Could this be because of a tighter sheet keeping more fines otherwise lost in the white water, resulting in a greater volume of fibers with a seeming reduction in other additives?

*Answer:* I do not believe so, since an examination of over 24 white water samples taken during a normal operation indicated that these gels were not keeping any more fines in the sheet.

*Question:* Are there any differences in the surface noted with the use of the manno-galactans?

*Answer:* It forms a flatter sheet, one that is more level.

*Question:* Do they modify the wet strength?

*Answer:* They seem to impart a very nominal type of strength to toweling.

*Question:* How do the manno-galactan gums compare with the ordinary starches in body?

*Answer:* One pound of the manno-galactan gum has as much body as ten pounds of starch in the same amount of water.

*Question:* What per cent of improvement in strength did you get in a sulphite sheet as compared with a groundwood sheet?

*Answer:* Generally speaking, the addition of starch has been uneconomical and ineffective. The manno-galactan gums are the first additives that has had a positive effect. In terms of Mullen strength, the optimum seems to be around 10%. Where starch does a good job, the efficiency ratio is around 5 to 1—that is, one pound of the manno-galactan gum will be doing the work of 5 pounds of starch.

***Meeting of February 10***

At the February 10 meeting Walter Couture of the Dilts Machine Co., Fulton, N. Y., described some of his impressions of the paper industry in England and other European countries, obtained during his recent trip.

# LETTERS TO THE EDITOR

## *Austrian Machinery Needs*

To the Editor *Tappi*

The attempts of the countries of Western Europe with regard to a consolidation of their economical situation have been generously promoted by the U.S.A.

In pursuance of the ideas of the ERP the Austrian paper industry made up a plan according to which it is intended to modernize and rationalize paper and pulp mills, and so to increase their profitableness. In that way the Austrian paper industry will contribute toward the aims of the Marshall-plan.

In order to realize the projects the financing of which will be effectuated by means of ERP funds, some of our member mills want to place certain orders in U.S.A.

The machinery to be purchased in the United States is listed as follows:

A. Complete plants
   (a) for the manufacture of newsprint (capacity of 100 metric tons per day)
   (b) for the manufacture of machine-made board (capacity of 60 to 70 metric tons per day)


**One of the Men behind Eastwood Wires**

Robert Zimmerman

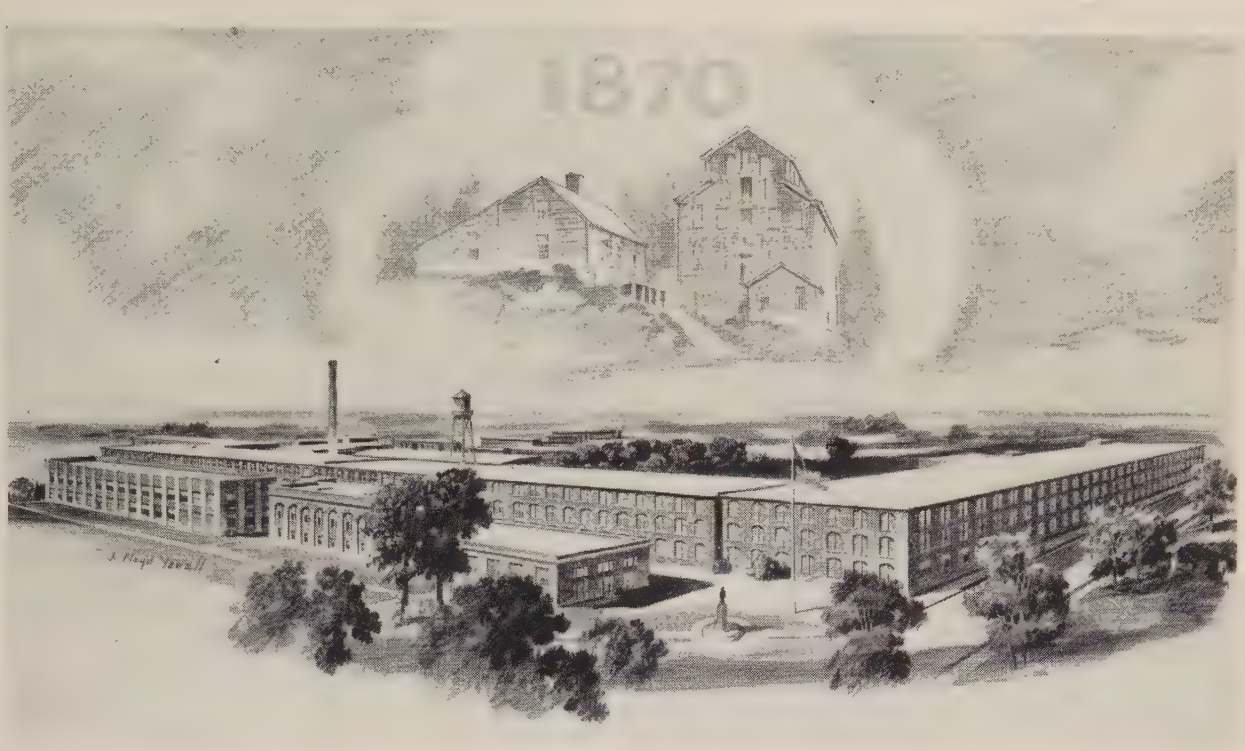
**"Seaming-ly" at Work**

The supervisor of our seaming department is shown operating a brazing machine across a fourdrinier wire, the ends of which previously had been prepared.

Under microscopic control, this machine brazes a fine, strong, uniform seam that probably will outlast the rest of the wire.

Seams once made by hand were a major source of complaints. Today—because of the brazing machine developed by the Eastwood organization—seam troubles have been largely eliminated.

**EASTWOOD-NEALLEY CORPORATION • Belleville, N. J.**



KENWOOD MILLS • 80th ANNIVERSARY • 1870-1950

# 80 Years of Paper Progress

Eighty years ago, the American paper industry was just beginning its great period of expansion. Since then paper, at lowering costs and in increasing volume, has changed the course and character of civilization, spreading culture and education, stimulating business, elevating the standard of living. The progress of the paper industry has paced the progress of America.

Also, just 80 years ago, F. C. Huyck & Sons began the manufacture of fine quality papermakers' felts. Recognizing that every paper problem is a felt problem, this organization has pioneered in research and in service, working always in the closest cooperation with the paper industry.

In observing its 80th Anniversary, F. C. Huyck & Sons looks forward to a continuation of the policy it has so long pursued, seeking new and better ways to help the paper industry produce more and better paper—the product most used by man.

*The dramatic story of paper is told in the sound-and-color film, "Paper — Pacemaker of Progress," and in a book under the same title. Both are presented by F. C. Huyck & Sons as a tribute to the Paper Industry. The book will be sent free upon request.*

**F. C. HUYCK & SONS • *Kenwood Mills* • RENSSELAER, N. Y.**

KENWOOD WOOL PRODUCTS

HUYCK *Felts*

(c) for the production of kraft pulp (capacity of 100 metric tons per day)

B. Separate machinery
(a) Newsprint machines (fourdriniers)
(b) Yankee machines for the manufacture of various MG papers
(c) Equipment for stock preparation, such as hydrapulpers, refiners, hydrafiners, pulpmasters, beaters, and Jordans
(d) Finishing equipment, such as calenders, cross-cutters, winders, and rewinders
(e) Equipment for the recovery of fibers (saveall)
(f) For the production of chemical pulp: modern Nichols-Freeman furnaces
(g) For digesting: digesters and modern equipment for indirect digesting, as well as for heat and sulphur dioxide recovery
(h) For screening: screens, knotters, and strainers
(i) For bleaching: modern equipment for bleaching plants
(j) For dewatering: modern wet machines
(k) Control devices for different stages of the pulp and paper production

We would highly appreciate your help in sending us names and addresses of U. S. manufacturers of the above listed machinery. If there are companies represented in Europe, please let us have the respective addresses.

*Ed. Note:* The above letter is printed for the benefit of any equipment builders who may wish to get in touch with the Austrian Paper, Pulp and Wood Association.

Vereinigung Österreichischer
Papier-, Zellulose-, Holzstoff-U. Pappenindustrieller
VI. gumpendorferstrasse 6
Vienna, Austria

# EMPLOYMENT SERVICE

Positions Open

P46-50. Machine designer. 10 or more years' experience on heavy machinery, preferably papermaking machinery. Apply by letter stating age, educational background, work experience. Machine builder in northeast.

P47-50. Mill superintendent experienced in tissue products including waxing, candy twisting, seat cover twisting, M.F. and creping tissues in white and colors. Two machine mills in New York State. Prefer man with chemical background and some knowledge of secondary fibers.

P48-50. Working superintendent. Paper converter. Knowledge of slitting and coating. Plant very near New York City.

P49-50. Manager for a new laminating division of a large Midwestern board mill. Must have knowledge of converting operations, sales experience and executive ability.

P50-50. Graduate physicist with one or two years' industrial experience, preferably with some training in chemistry, is required for development work involving the fields of mechanical properties of matter, heat and moisture transfer, optical properties of films, and sound absorption as applied to wood fiber products. Location: Upper Midwest.

P51-50. Midwest manufacturer is seeking sales representative for paper coatings, sizings, etc. Applicant should name experience in this field, a chemistry degree, laboratory experience Age 25–35.

P52-50. Large midwest commercial printer desires man with practical and technical experience in a mill making offset and tub sized printing papers.

Positions Wanted

E61-50. Research chemist. 45. Ph.D. Desires administrative technical position in progressive company in pulp and paper or related industry. 20 years' diversified research and development experience in sulphite and alkaline pulping, bleaching, papermaking, byproducts, and associated fields.

E62-50. Technical sales service position desired, especially with chemical or starch products companies. NYSCF Pulp and Paper Course graduate, 1941. Research chemist for large integrated book paper mill, adhesives chemist in multiwall bag factory, and chief chemist for fine paper specialties mill.

E63-50. Control or research chemist. 1936 Graduate Pulp and Paper Course, NYSCF. Extensive experience with insulating board and sanitary papers and toweling.


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Photo. Courtesy J. M. Huber Corporation


## Single Instrument Measures Smoothness, Porosity and Softness of Paper

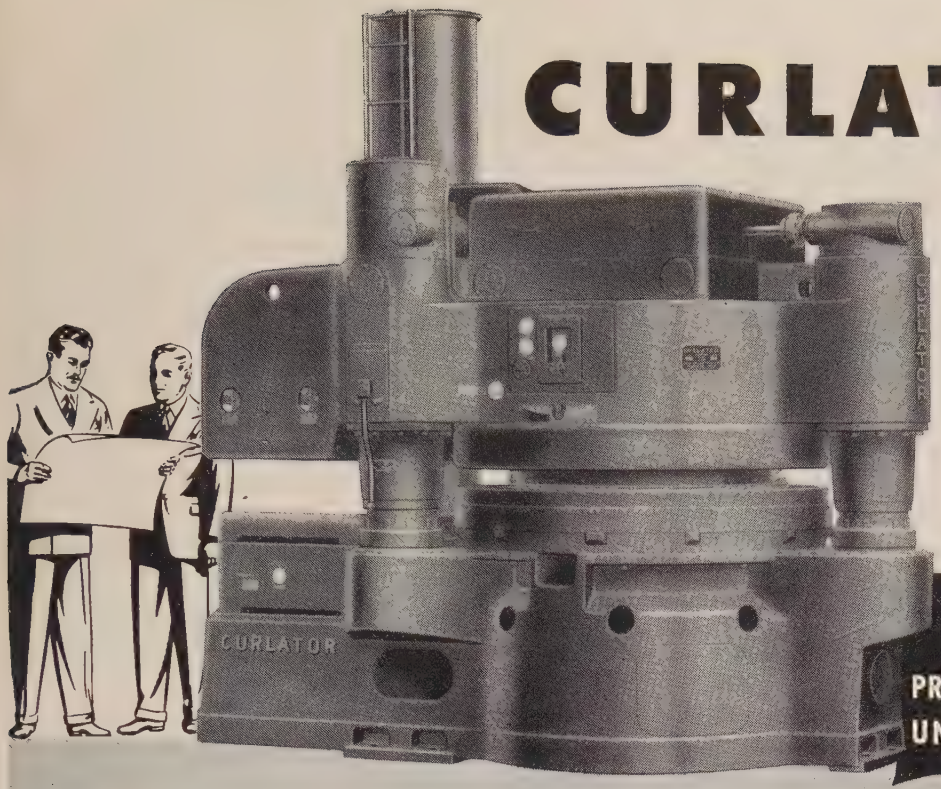
You just interchange the lower test plates in the GURLEY-HILL S-P-S Tester to convert it into an instrument for testing either smoothness, porosity or softness of any type of paper, by measuring a given air-leakage over or through the paper.

This 3 in 1 laboratory "assistant" is simple to operate, quick and accurate, and a real utility instrument where printing quality of paper must be reliably measured.

Bulletin No. 1400 describes the S-P-S Tester as well as other Gurley paper testing instruments. It's worth writing for today. W. & L. E. Gurley, Station Plaza & Fulton Sts., Troy, New York.

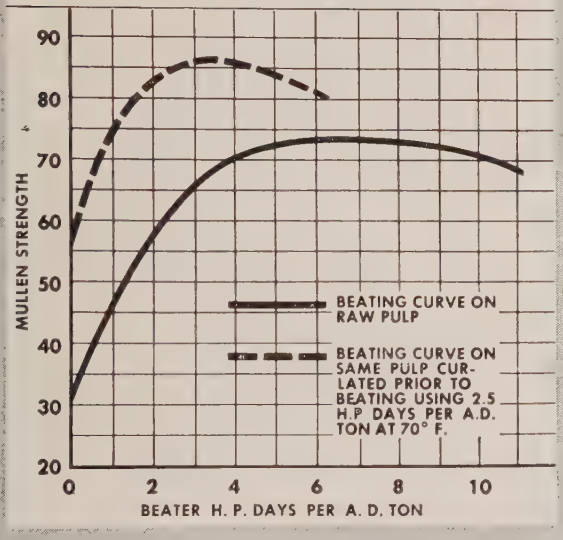
# CURLATION*

# New Process for Pulp Treatment




***Effect of Curlation Prior to Beating, on the Beatibility of***
**PREVIOUSLY DRIED NORTHERN UNBLEACHED SULPHITE PULP**

## MULLEN CURVES




## TEST DATA

| | HP/DT* | FREENESS | MULLEN | TEAR | DENSO-METER |
|---|---|---|---|---|---|
| RAW PULP | 0 | 843 | 32.4 | 127 | 4.2 |
| | 1 | 822 | 46.2 | 133 | 9 |
| | 2 | 813 | 55.0 | 114 | 14 |
| | 3 | 792 | 66.6 | 98 | 23 |
| | 5 | 723 | 71.8 | 87 | 68 |
| | 7 | 628 | 72.5 | 75 | 161 |
| | 9 | 548 | 73.6 | 71 | 454 |
| | 10 | 492 | 68.2 | 64 | 744 |
| | 11 | 413 | 69.7 | 64 | 1808 |
| | 14 | 240 | 63.5 | 54 | 4772 |
| PREVIOUSLY CURLATED PULP | | | | | |
| | 0 | 840 | 56.3 | 142 | 3.5 |
| | 1 | 805 | 74.5 | 116 | 15 |
| | 2 | 763 | 82.8 | 101 | 27 |
| | 3 | 690 | 86.0 | 97 | 106 |
| | 4 | 608 | 85.8 | 83 | 184 |
| | 5 | 562 | 80.8 | 74 | 544 |
| | 7 | 430 | 78.3 | 72 | 1612 |
| | 12 | 187 | 65.3 | 49 | 2880 |

*H. P. DAYS PER A. D. TON SPENT IN BEATING ONLY.

**The Curlator is a tool for altering the properties of pulp and paper to obtain qualities hitherto unachievable by mechanical means. The effects of curlation vary with the raw material and may be described only in relation to a particular pulp.**

**NOTE: Description of the testing procedures employed by the Curlator Corporation Laboratory will be provided upon request.**

• Illustrated here is the CURLATOR*—the machine that permanently curls and twists pulp fibres to produce improved sheet characteristics.

CURLATION is a fully tested and thoroughly proved mechanical process.

Its accomplishments in improved quality and outstanding savings justify the attention of paper mill executives responsible for sales and profits.

The chart shown here will be followed by others equally informative. Meanwhile, illustrated literature is yours for the asking.

**CURLATOR*** *Corporation*

565 BLOSSOM ROAD · ROCHESTER 10, NEW YORK

*T. M. Reg.—Curlator Corporation, Rochester, New York

# TALL OIL ABSTRACTS

## *With Particular Reference to Some End Uses*

The following abstracts are a continuation of those already published in *Tappi*. They were prepared by the National Southern Products Corp. (600 Fifth Ave., New York 20, N. Y.).

At the present time there are more than 40 industries who use tall oil for a variety of products. Tall oil is a product of sulphate pulp mills.

### *Adhesives*

**(1) Hellstroem, H., and Oehstroem, W. An adhesive for canvas and paper. Swedish patent 53,028 (May 1919).**

The adhesive is composed of 92 parts of asphalt, 5 parts of tall oil, and 3 parts of a siccative.

**(2) I. G. Farbenindustrie A.-G. Uses of tall oil in lacquer, adhesives, etc. German patent 517,902 (May 2, 1925).**

Purified tall oil is used as one of the constituents.

**(3) I. G. Farbenindustrie A.-G. An adhesive composition used for catching insects. British patent 306,906 (Feb. 26, 1929); B.C.A. 1930 B: 736; C. A. 23: 5286.**

Vegetable or animal oil (including tall oil), after chlorination at room temperature and then on a water bath, is mixed with resins, waxes, and machine oil.

**(4) Tengler, J. Rubber-containing varnish and adhesive. German patent 618,592 (April 5, 1932); Chem. Zentr. 1935, II: 3838.**

An unsaturated compound such as tall oil is vulcanized with rubber. The resulting product is then dissolved in organic solvents and again vulcanized. A dilution agent is used to produce the final desired consistency.

**(5) Holzapfel, M. Adhesive agent for paints. Norwegian patent 30,639 (March 5, 1918); Danish patent 24,399.**

A heated mixture of tall oil and a siccative is dissolved in a volatile solvent. The mixture may be heated with or without linseed oil.

**(6) I. G. Farbenindustrie A.-G. Tall oil adhesive. German patent 737,437 (Feb. 24, 1939).**

The adhesive is composed of either the vinyl esters of tall oil or the acids of tall oil stand oil and urea-formaldehyde condensation products.

**(7) Naamlooze Vennootschap Industrieele Maatschappij V. H. Nourry, Van der Lande. An adhesive and impregnating agent. Swedish patent 96,530 (May 23, 1938).**

Tall oil is mixed with isano oil and heated to a temperature above 150°C.

**(8) Peterson, M., and Son Aktieselskab. Adhesive and impregnating agents for paper and board. Norwegian patent 65,350 (April 1, 1940).**

The acids in tall oil are neutralized by heating the oil with a suspension of calcium oxide in water. Any excess lime acts as a filler. Most of the water is then evaporated, and the resulting product heated for 15 minutes at a temperature of 130–140°C.

**(9) Bent, Franklin A., and Peterson, Elbert A. The production and uses of tall oil ester resins. U. S. patent 2,424,047 (July 15, 1947); B.I.P.C. 17: 744; C. A. 41: 6418.**

The esterification of tall oil with polyhydric alcohols of aliphatic character produces resins having desirable film-forming properties. Some of the preferred alcohols are: allyl, methallyl, ethallyl, chlorallyl, etc. Direct esterification of the alcohol with the acid, with or without solvent, is one method of forming the product; the acid exchange between tall oil and a polymer of an allyl-type ester is another. Some of the uses of the products are: as adhesives and interlayers for laminated articles; as self-sustaining films; and for sizing and impregnating cloth felt, or paper.

**(10) Kirjakka, P. Preparation of a binding agent for use in paints and adhesives. Swedish patent 112,605 (Dec. 5, 1944); C. A. 40: 7658.**

Liquid resin, obtained from the production of sulphate cellulose, is heated with a drying oil for several hours at a temperature of 100–300°. Ester exchange, polymerization, and condensation take place during the heating period.

### *Amino Alcohol Esters*

**(1) Jayne, David W., Jr., and Day, Harold M. Production of tall oil esters of amino alcohol. U. S. Patent 2,322,202 (June 15, 1943); C. A. 38: 120.**

A mixture of tall oil, an organic sulphonic acid, such as benzenesulphonic or *p*-toluenesulphonic acid, and an amino alcohol with at least one primary or secondary group is heated to 150°C. to produce tall oil esters. Various reactants are listed.

### *Ammonium Salt*

**(1) Jayne, David W., Jr. Production of an ammoniated tall oil product. U. S. patent 2,238,021 (April 8, 1941); C. A. 35: 4335; B.C.P.A. 1942B, II: 234.**

Two parts of tall oil are dissolved in a small amount of alcohol; then anhydrous ammonia is introduced to prepare an alcoholic solution of anhydrous, completely saponified, ammonium tall oil soap. By maintaining a substantially anhydrous mixture, a relatively clear solution, containing a small excess of ammonia dissolved in alcohol, is obtained. The dark amber, homogenous fluid readily dissolves in water to give a nearly clear solution. Methanol, isopropanol, secondary and tertiary butanols, and tertiary amyl alcohol are the alcohols suggested for use. Anhydrous conditions are difficult to maintain when using ethanol. As a flotation agent the ammonium soap of tall oil has desirable properties. It is not commercially useful in the form of dilute aqueous solutions, because of the cost of shipping the large amounts of water present.

### *Asphalt Adhesive*

**(1) Hellstroem, H., and Oehstroem, W. An asphalt adhesive. Swedish patent 53,028 (May 17, 1919).**

An asphalt adhesive is composed of 92 parts of asphalt, 5 parts of tall oil, and 3 parts of a siccative.

### *Asphalt Emulsions*

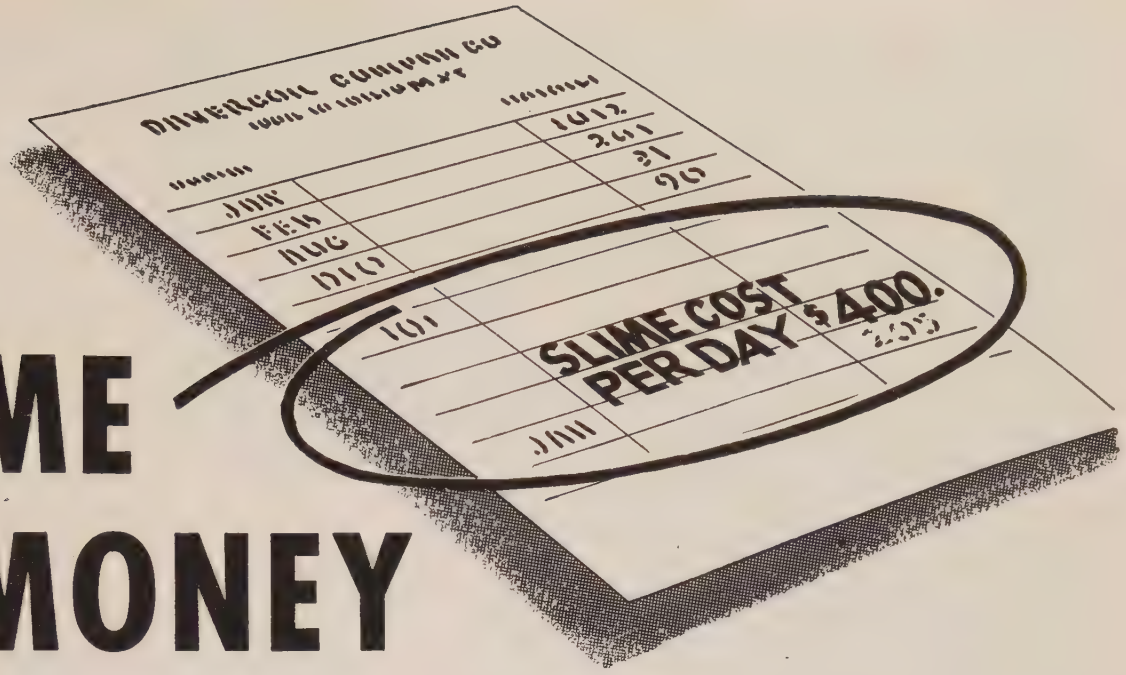
**(1) Cosenza, F. Asphalt emulsions. Italian patent 379,684 (Aug. 18, 1939).**

Asphalt is treated with tall oil as an emulsifier. The tall oil is heated to about 110°C., and 0.7% of tall oil is used. The tall oil-treated asphalt and half its weight of 0.4% NaOH solution are fed to a mixer and shaken for two to three minutes. A pretreatment, using 0.8% of the weight of asphalt of 80% $H_2SO_4$ at 110–130°C., is necessary when the asphalt has high ash content.

**(2) De Laney, William W. Asphalt emulsions. U. S. patent 2,350,548 (June 6, 1944).**

An emulsifier, used to produce intermediate breaking aqueous asphaltic emulsions, is used in two alkali metal soaps. In an alkali metal soap of petroleum hydrocarbon—insoluble pine-wood resin, from about 50 to 80% by weight of the soap is used. In an alkali metal soap of a material having a saponifiable acid constituent in the group, from about 20 to 50% by weight of soap is used. The acid constituent in the group consists of a mixture of fatty and rosin acids, which are the major constituents of tall oil.

**(3) Ebano Asphalt—Werke A.-G. Aqueous emulsions of bituminous materials. German patent 722,650 (April 24, 1940).**

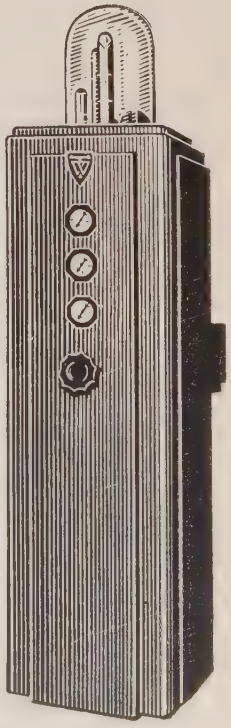



# SLIME IS MONEY

Although you may never see a bill, studies show that slime costs the average paper mill $400 or more a day and is responsible for such costly troubles as wet end breaks, pin holes, slime spots, poor sheet formation, etc.

Break-Point Chlorination, engineered to your particular needs, has proven its slime fighting ability in hundreds of installations. This process, applied by dependable W&T Chlorinators, to your fresh water supply, often in conjunction with chloramines or other corrective chemicals to your stock or white water, gets results because it destroys the very cause of the trouble — slime forming bacteria. Moreover, members of W&T's nation-wide service organization with over 35 years' experience at their fingertips, carefully engineer each installation, to insure that you will receive maximum protection against slime at the lowest possible cost.

Your nearest W&T Representative will be glad to give you full details on how Break-Point Chlorination can cut your slime costs. Call him today.



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I-28

A small amount of bitumen is mixed with tall oil and sulphur chloride. When the reaction is complete, more bitumen is added and the mixture emulsified with alkali.

**(4) Grader, Robert. Emulsions for bitumens. German patent 706,355 (April 17, 1941); addition to 703,599; C. A. 36: 2714.**

The emulsion may be made from tall oil or tall oil pitch. The oil or pitch is blown for 20–60 hours at 100–150° in the presence of cobalt resinate or copper naphthenate.

**(5) Rosencranse, Charles R. An asphaltic emulsion. U. S. patent 2,431,891 (Dec. 2, 1947); C. A. 42: 1734.**

Asphalt, which has been processed to penetration of 26–30 and a melting point of 210–260°F., is heated to 300°F. and run through a colloid mill in contact with an aqueous dispersion, at a temperature of 180°F. The aqueous dispersion is composed of: water 95.04%, petroleum-hydrocarbon-insoluble pinewood resin 3.0%, bentonite clay 1.4%, tall oil 0.10%, and caustic soda 0.46%. The final temperature of the emulsion is then rapidly reduced to 150°F. The sprayed colloidal-asphalt suspension is used to render coal dustproof, impervious to oxidation and moisture, and to prevent stacking and weathering of the coal.

**(6) Hjelte, Sixten Magnus. Bituminous emulsion. U. S. patent 2,434,243 (Jan. 13, 1948); C. A. 42: 2098.**

A suspension is prepared from water and the lime sludge obtained from the sulphate process of paper and pulp manufacture. An aqueous solution of alkali caseinate with a small amount of ammonia is then added, and this mixture is incorporated into a bituminous base, such as asphalt, tar and pitchy residues obtained from distillation or fractionating tall oil or liquid resin from waste liquors in the alkaline process of pulp and paper manufacture. A stable emulsion is obtained and adheres to damp and moist surfaces; therefore, it is useful for coating of walls and roofs. Upon drying, the surface is impermeable to water and resistant to temperature variations.

## *Axle Grease*

**(1) Keghel, M. de. The composition of tall oil and its utilization. Rev. Chem. ind. 35: 170–173, 202–206 (June, July, 1926); Pulp Paper Mag. Canada 26, no. 16: 529–531, 546, 548 (April 19, 1928); C. A. 21: 1354; T. S. 85: 212.**

Axle greases and dark colored printing inks can be manufactured from the resin fraction of tall oil.

## *Bibliography*

**(1) Bray, Mark W., Martin, J. S., and Smith, L. H. A list of references to literature on tall oil (tallol, liquid rosin, pine oil or black liquor soap). U. S. Dept. Agr., Forest Service, Forest Products Laboratory, Aug., 1938. R1129. 9p. Processed. 98 references.**

**(2) Jakobson, Torston. Tall oil or liquid rosin. Svensk Papperstidn. 42, no. 18: 473–474; no. 20: 513–514; no. 21: 534–535; no. 23: 600 (1939); no. 2: 24–25; no. 3: 44–45; no. 4: 65–66; no. 6: 109–110; no. 9: 175–179 (1940); 46, no. 6: 128–129, no. 183–186; no. 12: 283–284 (March 31, April 30, June 30, 1943); 47, no. 19: 473–474; no. 20: 492–494; no. 23: 580–581; no. 24: 605–607 (Oct. 15, 31, Dec. 15, 31, 1944); B.I.P.C. 10: 436.**

378 references.

## *Bitumen*

**(1) Johnson, James M. Use of amine soaps as surface-active agents. U. S. patent 2,419,404 (April 22, 1947); C. A. 41: 6070.**

The presence of small amounts of amine soap is desirable in bituminous paving material. The paving material, when amine soap is present, completely coats wet or dry aggregates and improves the adhesion and aging qualities. An acylamidoamine soap is dispersed in asphalt before use.

## *Bleaching*

**(1) Plauson, H. Bleaching soaps, fats, fatty acids and tall oil products. German patent 694,142 (Oct. 26, 1938); C. A. 35: 5338.**

An alkali—or formaldehyde—hydrosulphite and hydrogen peroxide in the amount of 0.5–4% of the material to be bleached are used in the presence of 10–60% of water. The ratio of the hydrosulphite to hydrogen peroxide is from 1:3 to 1:6. Sulfuric acid is added when bleaching fatty acids or oils.

## *Board*

**(1) Bonell, F. T. B. Impregnation of paper and boards. Swedish patent 109,319 (Oct. 14, 1943); Chem. Zentr. 1944, II: 386.**

Calcium salts of tall oil acids, which have been purified by an oxidation treatment or treated with sulphur chlorides, are used to impregnate paper or board.

**(2) King, George D. A pressed fibrous product. Canadian patent 434,183 (April 16, 1946).**

An aqueous emulsion containing tall oil is prepared by heating the oil above 140°F. This emulsion is added to a fibrous material, the tall oil is precipitated into the fibers by an acid-reacting metallic salt, and the suspension formed into a coherent mass, dried and consolidated at a temperature between 350° and 500°F. under a pressure of 500 to 1500 p.s.i. The hardboard thus produced contains 2–10% tall oil, has a density of 50–72 pounds per cubic foot, and varies in thickness from 0.1 to 0.25 inch.

**(3) Bergstrom, Hilding O. V., Heijmer, Gustaf B., and Trobeck, Karl G. An oil-hardened fiberboard. U. S. patent 2,437,931 (March 16, 1948); C. A. 42: 5226.**

An impregnating oil is obtained by the distillation of tall oil under vacuum. The fraction up to the point at which all the tall resins and part of the fat acids have been distilled is collected. This fraction may be used, with 10 parts of its weight of linseed oil and twice its weight of water, to form an emulsion which is stable for at least 24 hours. Upon heating the impregnated boards, the oil drys and hardens causing a hardening of the board.

## *Briquetting Agent*

**(1) Lange, A. Use of rosin soap from the soda process in the manufacture of briquets. German patent 314,445 (April 30, 1918); Chem. Zentr. 1919, 4: 927.**

Tall oil soap is separated from black liquor, warmed with sodium hydroxide, and the large soap flakes treated with sodium peroxide. After the solution has been evaporated and the excess soda crystallized, the liquid may be used as a binding agent in the manufacture of briquets.

## *Cement*

**(1) Lidvall, N. A. F. Tall oil in cement, Swedish patent 82,448 (Jan. 22, 1935); C. A. 31: 2771.**

Sulphate oil or sulphate oil pitch may be mixed with cement.

**(2) Lidvall, N. A. F. Sulphate oil as a binding media. Swedish patent 85,020 (Dec. 3, 1935); C. A. 30: 6478.**

The sulphate oil is converted into a stable form by heating with calcium hydroxide, cement, or another stabilizing agent. More stabilizing agent is added until a product of the desired hardness is obtained. Sand, stones, fibers, and mineral oils may be added as filling materials.

**(3) Chemische Fabrik Grunau, Landshoff und Meyer A.-G. Use of tall oil in cement. German patent 681,856 (Feb. 7, 1936).**

A cement may be made of a solution of tall oil in benzene, benzine, hydrocarbons, trichloroethylene or similar solvents, fillers, and vaseline.

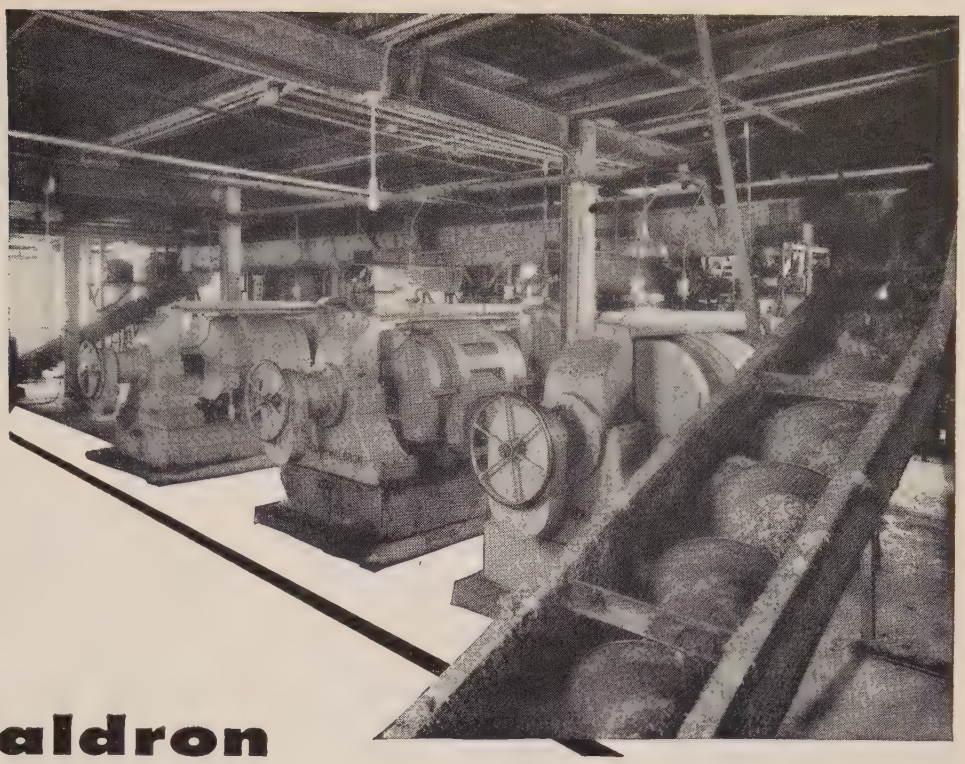
**(4) Holzapfel, M. Preparation of a cement from tall oil. Norwegian patent 30,239 (March 18, 1918).**

Tall oil is heated with lime then mixed with cement. The product can be used as a cement, an adhesive for dyes, or as a general adhesive.

**(5) Booth, Robert Ben. Increasing the plasticity of Portland cement mixtures. U. S. patent 2,311,289 (Feb. 16, 1943).**

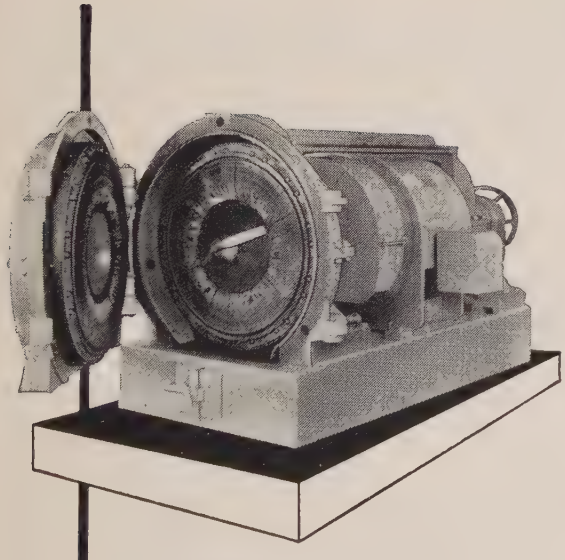
Black liquor soap, added prior to, during mixing, or incor-

# *Mills that compare* **Refiners** *choose* **Sprout-Waldron**



**The MAIZEWOOD Installation**

**A battery of S/W Refiners and stock conveyors recently installed at the Maizewood Insulation Co., Dubuque, Iowa.**



S/W Refiners do a wide variety of jobs—all of them thoroughly and economically. Here are some applications: refining kraft, soda, and sulphite knotter and fine screen rejects; hogged bull screen rejects; knotter and second screen rejects of raw groundwood; *semi-chemical chips of all kinds;* spent chips after extraction process; bagasse, straw, and similar grasses; breaking down lumps in reclaimed waste paper stock; reduction and refining of rag and other half stocks, etc., etc.

When all the pros and cons are weighed about pulp refiners, your choice will be Sprout-Waldron. Here is a high quality refiner that does more at less cost. A humdinger in any type of pulping, it far outperforms similar equipment in semi-chemical operations.

Its rugged construction, precision engineered, includes the unique peripheral control ring feature. This provides great flexibility of adjustment, enabling you to produce a wide variety of pulp characteristics. **With the Sprout-Waldron you can pinpoint exact pulp requirements.** Long-life plates are available in many styles . . . are easily changed and inexpensive.

Your initial investment in a S/W Refiner is comparatively low. High production rates, economy in power consumption, ease of operation, adjustment, and maintenance, mean additional savings.

Let a Sprout, Waldron representative explain how these refiners can step up your output and cut operating costs, or ask for Bulletin R-748. Address Sprout, Waldron & Co., Inc., 38 Waldron Street, Muncy, Penna.

**Sprout, Waldron**

*Manufacturing Engineers*

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112

porated during grinding of the clinker, increases the plasticity of Portland cement. From 0.001 to 0.01% of black liquor soap, based on the amount of dry cement in the wet mix is added. To obtain a more uniform distribution in the mix the soap is mixed with fine sand or kieselguhr.

**(6) Mark, Jacob G. Scale resistant concrete and cement. U. S. patent 2,240,144 (May 6, 1947); B.I.P.C. 17: 606; C. A. 41: 5278.**

Used as grinding aids and air entraining agents in Portland cements for concrete.

## *Cement Dye*

**(1) Kroenlein, J. Liquid rosin in cement dye. Swedish patent 46,968 (April 25, 1918).**

Liquid rosin can be used as a component of cement dye.

## *Cleaning Agent*

**(1) Lowe, Charles S. Emulsion cleaner. U. S. patent 2,374,133 (April 17, 1945).**

A solvent emulsion cleaner contains (parts by weight) 40–60 of tall oil, 7.6–11.4 of triethanolamine, 20–30 of 50% potash solution, and 6.4–9.6 of pine oil. Sufficient potash is used to insure an excess after reacting with all the tall oil in the mixture.

**(2) Unger, Th. B. Preparation of cleaning agents from tall oil. Norwegian patent 65,134 (Dec. 21, 1940).**

Crude tall oil is dehydrated and sulphonated. It is treated with hydrogen peroxide at 80–90°C. to improve the odor, then neutralized. The neutral product can be mixed with other soaps, and the mixture dried.

## *Coating Agent*

**(1) Getty, Edmund. The use of sulphonated tall oil in the manufacture of coated paper. Paper Trade J. 114, no. 8: 130–132 (Feb. 19, 1942); B.I.P.C. 12: 215.**

Excellent results in coating application and in the quality of the finishing paper have been obtained from the use of sulphonated tall oil in coating mixes. When used in relatively small quantities, sulphonated tall oil gives better brushing and a more desirable surface appearance than does sulphonated castor oil. Using large quantities of sulphonated tall oil increases the folding endurance and bursting strength of the test sheets. High tear resistance is imparted to test sheets with clay-casein coatings containing sulphonated tall oil.

**(2) Stewart, C. W. An adhesive for paper clay coatings. Pulp Paper Mag. Canada 43, no. 4: 333 (March, 1942).**

The formula for a brush-type coating is as follows: zein 10, sulphonated tall oil 4, caustic soda 0.4, water 125, and china clay 100 parts.

**(3) Beckman, Arnold O., Bush, William A., and Rubin, Harry. Coating material and product. U. S. patent 2,277,287 (March 24, 1942).**

Two coatings, one a tacky undercoat and the other a non-tacky overcoat, are proposed in the coating of paper. A mixture of tall oil and castor oil may be used for the first coat.

**(4) Grohn, Hans, and Herminghaus, Heinrich. Compositions of coatings. German patent 632,084 (July 2, 1936); C. A. 30: 6969.**

A solution of organic colloids, such as copal, factice, or rubber, and tall is treated with a drier. The solution is then mixed with fillers, pigments and drying oils, or diluted with an organic solvent.

**(5) Rice, Williams J. Method of making a coating composition. U. S. patent 2,373,709 (April 17, 1945).**

A mixture of tall oil, rosin, and lime in an amount of 10–15% of the rosin used gives a resinous reaction product. It is an ungelled, hard resin, and is soluble in organic solvents.

**(6) Stewart, Charles W. Tall oil in papercoating. U. S. patent 2,370,268 (Feb. 27,1945).**

The composition of a coating is given as follows: 63% sulphonated tall oil 2–8, zein 6–16, sodium hydroxide 0.3–0.5, water 60–200, China clay 100 parts. Some of the advantages of this coating are its light color, elimination of foaming difficulties, improved printing characteristics, and ease of use in connection with a roll-coating machine. Another coating contains zein, tall oil, potassium rosin size made from hydrogenated rosin, sodium hydroxide, water and China clay; another contains tall oil, zein, urea, ammonium hydroxide, water and China clay.

**(7) Kemner, H. Use of tall oil in the coating industry. Farben, Lacke, Anstrichstoffe 1, 20–21 (1947).**

Good coatings can be obtained by the treatment of vacuum-distilled tall oil with calcium or zinc oxide then esterification with suitable polyhydric alcohols; by incorporation into alkyd resins; or by reaction with China wood or oiticia oil, then esterification.

**(8) Hjelte, Sixten Magnus. Bituminous emulsions. *Loc. cit.*, Asphalt Emulsions (6).**

## *Core Binder*

**(1) Imperial Chemical Industries, Ltd., and Bunbury, H. M. Tall oil in core binders. British patent 348,315 (March 29, 1930); C. A. 26: 3081.**

Either tall oil or liquid rosin is used with or without linseed oil, glue, starch or alkali. Cobalt linoleate or alkali may be used as an emulsifier.

## *Cotton Bucking*

**(1) Kollmann, Ieo. Tall oil in textile processing. Melliand Textilber. 20, no. 2: 129–131 (Feb., 1939); C. A. 33: 5666; B.I.P.C. 9: 368.**

A soap product for use in bucking cotton, degumming silk, scouring wool, desizing rayon, and in dyeing processes is produced by the neutralization of tall oil with caustic alkali.

## *Cutting Oils*

**(1) Keghel, M. de. Tall oil in cutting oils. Rev. Chim. ind. 35: 170–173, 202–206 (June, July, 1926); Pulp Paper Mag. Canada 26, no. 16: 529–531, 546, 548 (April 19, 1928); C. A. 21: 1354; T. S. 85: 212.**

Distilled fatty acids of tall oil may be used as a base for cutting oils, textile oils, leather oils, etc. Listed are a number of uses that can be made of the distilled fatty acids, the resin fraction, and the sulphonated resin acids.

**(2) Kleine, C. W. A. Lubricating and boring oils. Swedish patent 48,619 (Sept. 24, 1917).**

Liquid rosin is treated with alkali, in such a manner that an acid salt is not formed, then distilled using superheated steam.

**(3) Obst, W. Utilization of tall oil. Seifensieder-Ztg. 60: 856 (1933).**

This is a description of methods of separating the fatty and rosin acids in tall oil and of deodorizing tall oil soap. Listed are a number of uses of tall oil of which cutting oils is one.

**(4) Patch, Richard H., and Dambacher, Fritz. Product resulting from polymerization, condensing and oxidizing tall oil. U. S. patent 1,938,532 (Dec. 5, 1933); C. A. 28: 1207; B.C.A. 1934B: 848.**

A dark, clear oil, nonsludging and practically odorless is produced from crude tall oil by the action of oxygen on the oil at a temperature of 110°C. for about 40 hours. The oil can be used as a cooling oil in cutting metals. The specific gravity and viscosity of the oil may be varied by using different catalysts.

**(5) Patch, Richard H., and Leaper, Joseph M. Cutting oil for metal. U. S. patent 1,919,125 (July 18, 1933); C. A. 27: 4919.**

A number of formulas are given for the preparation of cutting oils. Some of the formulas are as follows: tall oil 5%, lard oil 5%, and mineral oil 90%; tall oil 8%, oleic acid 6%, 35 deg. caustic soda solution 4%, monoethyl or monobutyl ethylene glycol 4%, mineral oil 78%; tall oil 25%, mineral oil 75%.

**(6) Abramowitz, William L., and Beach, Ralph M. Cutting oils from sulphurization of oils and fats. U. S. patent 2,313,611 (March 9, 1943); C. A. 37: 5266; B.C.P.A. 1944B, II: 361.**

Sulphurization of oils and fats with liquid sulphur halides for about $^1/_2$ hour in the presence of ammonia produces a

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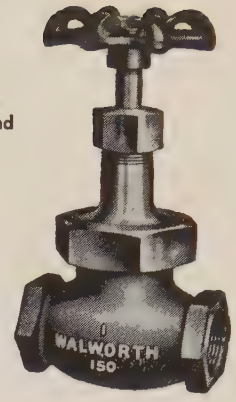


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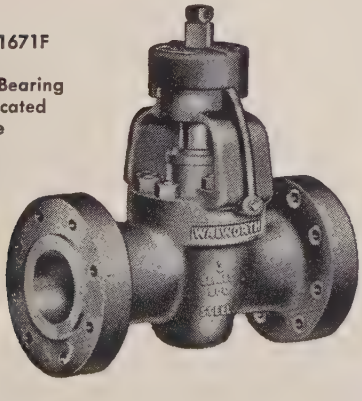


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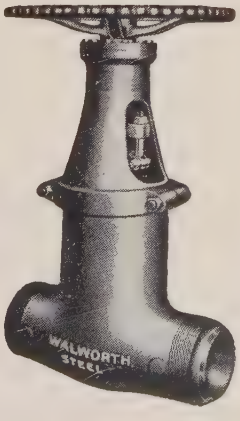


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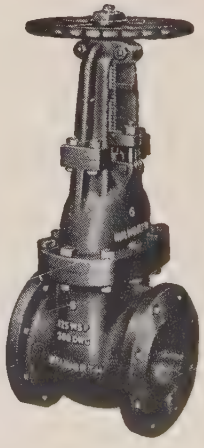

material useful for the manufacture of cutting oils. The liquid sulphur halides suggested for use were: thionyl chloride, sulphur dibromide, sulphuryl chloride, and sulphur mono- or dichloride. The methyl esters of tall oil fatty acids were among the products used.

**(7) Pollak, Arthur, and Hastings, Randall. Sulphurized cutting oils. U. S. patent 2,198,562 (April 23, 1940); C. A. 34: 6444.**

Tall oil is desirable in cutting oils because of its sulphur-retaining capacity. Refined tall oil will absorb in excess of 25% by weight of sulphur, which is greater than that of either mineral or vegetable oil. Upon standing, the oils deposit a sludge, but this may be reduced to a minimum by heating the tall oil and sulphur under controlled conditions. An example of the process is given.

**(8) Sudholz, Louis H., and Flemming, Cecil D. An improved cutting oil. U. S. patent 2,429,198 (Oct. 14, 1948); C. A. 42: 359.**

The deposition of sludge from a cutting oil, which is composed of sulphurized tall oil or mineral oil, can be reduced to a small amount of a nonadhesive material by the addition of 5–30% of an aromatic solvent derived from high-temperature catalytic conversion of petroleum oils.

## *Degreasing Agent*

**(1) Curran, Alton F. Liquid degreasing agent. U. S. patent 2,107,287 (Feb. 8, 1938); C. A. 32: 2655, T. S. 109: 47.**

Tall oil is saponified with potassium hydroxide or a 50–50 mixture of potassium and sodium hydroxides. A tall oil containing 50–70% rosin acids, 20–40% fatty acids, and about 10% unsaponifiable matter is preferred. The alkali solution is sprayed into the tall oil at room temperature, 25–75% of liquid petroleum hydrocarbon is added as a grease solvent, and the emulsion is stabilized with 15–30% cresylic acid.

**(2) Curran, A. F. Composition of a liquid degreasing agent. U. S. patent 2,107,288 (Feb. 8, 1938); C. A. 32: 2655; T. S. 109: 47.**

The degreasing composition is made by mixing tall oil and an anhydrous liquid solvent, such as cresylic acid. The resulting composition is diluted with a liquid hydrocarbon grease solvent such as carbon tetrachloride or kerosene.

**(3) Hodges, Paul. Degreasing agents. U. S. patent 1,993,098 (March 5, 1935); C. A. 29: 2632; T. S. 101: 297.**

Examples are given of the compositions of various degreasing agents containing floating soap. An example of one is: floating soap 25%, alcohol 37.5%, trichlorethylene 37.5%. Floors may be scrubbed with the compositions, or hides and metal articles may be degreased by dipping.

## *Deinking*

**(1) Schmid, W. Utilization of liquid rosin. Zellstoff u. Papier 13, no. 12: 571–572 (Dec. 1933); B.I.P.C. 4: 100; C. A. 28: 2529.**

Liquid rosin obtained from tall oil is used for deinking of newsprint.

## *Detergents*

**(1) Bennet (Hyde) Ltd. Detergents. British patent 491,960 (Sept. 12, 1938); C. A. 33: 1419.**

When tall oil is saponified with an excess of an alkaline solution, a grease solvent added, and phenol added in amount sufficient to neutralize excess alkali; a stable degreasing agent is obtained.

**(2) Friesenhahn, P. Washing, wetting, and emulsifying agent. Swiss patent 152,220 (May 31, 1930).**

The agent is a mixture of sulphonated organic compounds and alkali phosphates. Sulphonated tall oil produces a desirable agent. Some of the uses are: as carrier for insecticides, in the preparation of cold asphalt mixtures for briquetting dust, and as a soap in the textile industry.

**(3) Dreger, Emil E., and Ross, John. Process for the production of a detergent. U. S. patent 2,290,583 (July 21, 1942); C. A. 37: 483.**

Fatty acids of tall oil are chlorinated with a stream of chlorine, either directly or in a solvent. Five parts of glycerol are added to 55 parts of 20% oleum at a temperature of 10°C. The temperature is increased to 30°C. and 20.5 parts of the chlorinated tall oil added slowly to the glycerol-oleum mixture. The acid mixture is agitated for two hours, poured into 250 parts of water and 250 parts of ice, then neutralized with approximately 180 parts of 23.6% sodium hydroxide. It is used in compositions to be used as dispersing, emulsifying, penetrating, solubilizing, washing, and wetting agents.

**(4) Garverich, Edwin S., and Martin, William L. Preparation of a detergent agent. U. S. patent 2,411,090 (Nov. 12, 1946).**

The composition of a detergent agent is as follows: 5–25% tall oil, 25–75% bentonite, about 5% alkali metal phosphate, and 25–45% alkali metal sub-silicate.

## *Disinfectant*

**(1) Hyde, Elmer H. Composition of a disinfectant. U. S. patent 1,882,618 (Oct. 11, 1932); C. A. 27: 810.**

Tall oil is mixed with water-gas-tar distillate, water, and a liquid resinate. The distillate is obtained from water-gas-tar between 100 and 335°C., and the liquid resinate contains rosin and fatty acids, lignin, alkalies, and water. Examples of compositions are given.

## *Drawing Oil*

**(1) Nill, Edward A. Method of making a drawing composition. U. S. patent 2,326,387 (Aug. 10, 1943); C. A. 38: 541.**

A sulphonated mixture of either crude or refined tall oil and sperm oil is used to make a drawing oil. The mixture contains 10–30% sperm oil, which plasticizes the tall oil and dries the product to a softer film.

## *Dyeing*

**(1) Kollmann, Leo. Tall oil in textile processing. Melliand Textilber. 20, no. 2: 129–131 (Feb., 1939); C. A. 33: 5666; B.I.P.C. 9: 368.**

A soap, useful in degumming silk, scouring wool, bucking cotton, desizing rayon, and in various dyeing processes, is made by the neutralization of tall oil with caustic soda.

## *Emulsifying Agent*

**(1) Becher, C., Jr. Tall oil or liquid rosin. Chem.-Ztg. 60, no. 37: 373–375 (May 6, 1936); T. S. 103: 246; B.I.P.C. 6: 415.**

Tall oil consists of fatty (oleic and linolenic, with small amounts of palmitic and isolinolenic acids) and rosin acids (oxyresinic and abetinic acids). Methods of purification and the use of tall oil for soaps and asphalt emulsions are discussed.

**(2) Bjorn, E. A. V. Emulsifier for the preparation of emulsions for road surfacing. Swedish patent 72,-188 (Oct. 21, 1929); T. S. 94: 219.**

The emulsifier is composed of hydrolyzed and polymerized resin acids and sulphonated rosin acids, or other materials having strong moistening properties.

**(3) Boehme, A.-G., H. Th. Capillary active ester-like sulphuric acid compounds. German patent 596,510 (June 19, 1930); C. A. 28: 5185.**

Tall oil is treated with hydroxylated or hydrogenated derivatives of ethyl sulphite or ethionic acid. The resulting products are used as cleaning, dispersing, foam-forming, or wetting agents.

**(4) Chem. Fabrik Floersheim Dr. H. Nordlinger A.-G. A substitute for Turkey-red oil. German patent 310,541 (Dec. 15, 1915); 314,017 (Dec. 19, 1915); J. Soc. Chem. Ind. 39: 164A; Pulp Paper Mag. Canada 19: 540.**

A product resembling a fatty oil and a solid acid resembling abietic acid are obtained by the distillation of liquid resin under reduced pressure. The distillate, after separation of any crystallizable constituents, is sulphonated with sulphuric acid; and the sulphonic acids, which are almost completely water soluble, can be almost completely neutralized. Semi-solid or gelatinous soaps result from complete neutralization of the acids. The solutions can be used in the textile industry for dressing and mordanting and as a media for the emulsification of oils, fats, and organic liquids with water.

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**(5) Disney, John H., and Kernot C. Emulsions of bituminous substances of oils. British patent 274,-142 (Jan. 15, 1926); French patent 628,302 (Nov. 26, 1926); C. A. 22: 2017; Chem. Zentr. 1928, 1: 452.**

A product, which may be used in liquid form or dried to a solid and prepared for use when desired by adding dilute alkali, is made by mixing one part of dry gelatin in one part of water with two parts of liquid rosin. It may be used for emulsifying coal tar, pitch, or mineral, vegetable, or similar oils.

**(6) Grader, Robert. Bituminous emulsions. German patent 703,599 (Feb. 6, 1941); C. A. 36: 652.**

Tall oil, containing 15–28% chlorine, is used as an emulsifier for bitumen. The chlorinated tall oil is added to bitumen, heated to 100–120°C., and the hot bitumen is transferred to an aqueous alkaline solution and agitated until the mixture is emulsified.

**(7) Hein & Company. Aqueous emulsions of tall oil. German patent 479,085 (June 10, 1925); C. A. 23: 4842.**

The dispersion is made with aniline or its homologs, in the presence of soap with or without naphthol.

**(8) Hodges, Paul. The use of floating soap and its derivatives. U. S. patent 1,992,949 (March 5, 1935); T. S. 102: 168; C. A. 29: 2497.**

Floating soap may be used as an emulsifying agent for casein, linseed oil, lubricating oil, paraffin wax, pine oil, rosin, rubber, stearic acid, and turpentine. A paraffin emulsion is made by melting together 10 parts of soap, 10 parts of wax, and 3 parts of oleic acid. This molten mixture and 50 parts of water produce the emulsion. It is also useful in the flotation of sulphide and oxide minerals and in producing suspensions of pigments.

**(9) I. G. Farbenindustrie A.-G. Tall oil derivatives as dispersing agents. British patent 340,272 (July 19, 1929); C. A. 25: 4136; B.C.A. 1931B: 334 .**

True sulphonic acids are produced by treating tall oil esters or amines with sulphonating agents. The products may be used in acid or alkaline liquids or those containing alkaline earth in carbonizing, dressing, dyeing, mercerizing, and milling processes.

**(10) Imperial Chemical Industries, Ltd., Bunbury, H. M., and McGlynn, R. P. Emulsifying agents. British patent 369,985 (Nov. 29, 1930); C. A. 27: 3046.**

Tall oil or liquid rosin is heated with a polyhydric alcohol, in the presence of an esterification agent such as calcium or magnesium glycerate, and the resulting ester is treated with a sulphonating agent at a temperature not greater than 40°C. to form a sulphuric ester.

**(11) Meyer, Erwin, and Uddeholms Aktiebolag. Aqueous emulsions. British patent 365,844 (Feb. 25, 1931); C. A. 27: 1687.**

Turpentine oil, preferably obtained from the sulphate process or charcoal industries, is used as the sole emulsifying agent in the preparation of aqueous emulsions. Colophony, liquid rosin, paraffin, and bitumen-asphalt emulsified with water may be used for impregnating wood, paper, textiles, and sizing paper.

**(12) Nuessel, Hans. Bituminous emulsions. Bitumen 11: 45–51 (1941); Chem. Met. Eng. 49, no. 1: 156–157.**

The behavior of an asphalt, when dispersed in potassium hydroxide and the dispersion promoted by the addition of 1, 2, and 3% of tall oil, is shown graphically. Desired fineness is not approached until higher than 3% concentrations of alkali are reached.

**(13) Obst, W. New uses of tall oil. Seifensieder-ztg. 60: 856 (1933).**

Methods of deodorizing soap containing tall oil and of separating the rosin and fatty acids are given. Tall oil may be used in the manufacture of cutting oils, emulsifying agents, factice, plastic masses, road emulsions, and textile oils.

**(14) Pollak, Arthur. Utilization of tall oil. Oil & Soap 17: 87–89 (1940); C. A. 34: 3492; B.I.P.C. 11: 32; B.C.P.A. 1940B: 544.**

Tall oil is used in making asphalt emulsions, as a substitute for fatty acids in softening rubber, in the soap industry, and in the petroleum and paint industry.

**(15) Thompson, C. H., and McGivern, W. J. Bituminous emulsions. British patent 263,307 (Jan. 5, 1926); C. A. 22: 163.**

Liquid rosin is used with a natural bitumen and an alkali as an emulsifying agent.

**(16) Uddeholms A.-B. The preparation of emulsions. Swedish patent 71,267 (Oct. 31, 1928).**

Tall oil is mixed with water to prepare an emulsifying agent to be used with turpentine oil.

**(17) The uses of tall oil. Tech. Bull. Paper Makers' Assoc. Gt. Britain Ireland 13, no. 8: 109 (Aug., 1936); B.I.P.C. 7: 29; T. S. 103: 340.**

Refined tall oil is used in the manufacture of hard and semi-hard soaps. These soaps have good lathering and emulsifying properties. Tall oil is also used in asphaltic emulsions, fruit tree sprays, and varnishes for outdoor use.

**(18) The use of waste products. Farbe u. Lack 1927: 116, 127; Chem. Zentr. 1927, 1: 2492.**

The uses of tall oil include the preparation of emulsions, impregnating agents, and substitutes for rosin oil.

**(19) Allgemeine-Elektricitats-Ges. Preparation of a paste from solid polyvinyl compounds. German patent 642,751 (Dec. 20, 1932).**

Tall oil, which has been saponified with potassium carbonate, can be used as an emulsifying agent in the preparation of pastes of solid polyvinyl compounds. The compounds can be diluted with water, and the emulsions can be used for coating compounds.

**(20) Alsmark, Kurt O. Oil-in-water type emulsions. U. S. patent 2,359,503 (Oct. 3, 1944); C. A. 39: 827.**

An oily material, such as tall oil, tall oil acid, animal or vegetable oil, or mixtures of these; after its acid value has been adjusted to 70; is heated to 100–110°C. and treated with about 60% of the total quantity of aqueous alkali solution required to neutralize all the free acidity. (The alkali solution is added at the temperature of the oil.) After the addition of a suitable quantity of softened water at 80°C. and a small quantity of disodium phosphate, the solution is cooled to 50–55°C. and enough alkali to neutralize the acidity is added. The product is a stable emulsion and is used as a cooling lubricant for metal-cutting tools.

**(21) Alsmark, Kurt O. The preparation of lubricants. Swedish patent 107,962 (Feb. 11, 1942).**

Liquid rosin is first warmed to 85°C. in the presence of soft water; then treated with an aqueous solution of potassium hydroxide, which contains disodium phosphate. The resulting emulsion is warmed at 92–95°C. for 15 minutes with stirring.

**(22) Alsmark, Kurt O. Preparation of oil-in-water type emulsions. Swedish patent 108,980 (May 30, 1942).**

The acid number of tall oil is adjusted to about 80 by a cracking treatment, and the saponification is carried out in two steps. The first step is with potassium hydroxide at 100°C., water is added and further saponification is carried out at 50°C. A small amount of disodium phosphate is added during saponification.

**(23) Alsmark, Kurt O. Preparation of oil-in-water type emulsions. Swedish patent 110,291 (April 6, 1943); addition to 108,980.**

The process described in patent 108,980 is applied to a mixture of mineral oils or tar oils with fatty acids or vegetable or mineral oils. Also, in this process the acid number is adjusted to 70.

**(24) Alsmark, Kurt O. Preparation of oil-in-water type emulsions. Swedish patent 111,197 (Oct. 7, 1943); addition to 108,980.**

In this process the water added during the saponification steps contains a small amount of sodium nitrate and an agent for protection against corrosion.

**(25) Basset, H., and Szidon, V. The preparation of emulsions. French patent 630,168 (March 4, 1927).**

A mixture of tall oil and asphalt, tar, and coal tar is treated with enough alkali to saponify the tall oil.

**(26) Cosenza, F. Asphalt emulsions. *Loc. cit.*, Asphalt Emulsions (1).**

**(27) Ebano Asphalt-Werke A.-G. Aqueous emulsions of bituminous materials. *Loc cit.*, Asphalt Emulsions (3).**

**(28) Oranienburger Chemische Fabrik A.-G. Preparation of cleaning, emulsifying, or other agents. German patent 582,790 (Jan. 14, 1927; Aug. 23, 1933); C. A. 28: 923.**

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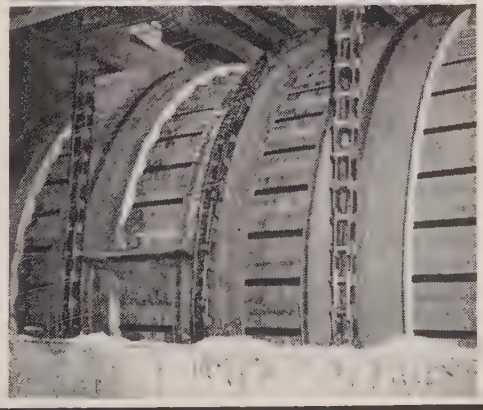

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An agent; which may be used for cleaning, emulsifying, and wetting; can be prepared by treating natural or synthetic fats, fatty acids, (tall oil is mentioned) or fat-like substances with ketones, alcohols, or lactones. Sulphuric acid halogenhydrins are used as water-removing agents.

## *Breaking of Emulsions*

**(1) Arguss, Meyer S., and Schindler, Hans. The breaking of crude oil emulsions. U. S. patent 2,365,853 (Dec. 26, 1944).**

The efficiency of the alkali salts of sulphonated tall oil, used as demulsifying agents, may be improved by using them with an alkali salt of oil-soluble sulphonic acids derived from petroleum oils by treatment with sulphuric acid. It has been found advantageous to also add a small proportion of a surface-active material.

**(2) Arguss, Meyer S., and Schindler, Hans. Process of demulsifying oils. U. S. patent 2,365,852 (Dec. 26, 1944).**

Typical formulas for demulsifying agents are given. In one formula 90% of sulphonated tall oil, 6% of glycerol dichlorohydrin, and 4% of N-butylaniline were used. The demulsifying agent was used in a ratio of 3 gallons to 120 barrels of oil. Other amines and substitutes for glycerol dichlorohydrin were mentioned.

## *Explosives*

**(1) Nitroglycerin A. B. Explosive constituent. Swedish patent 42,470 (March 24, 1916).**

Nitrated liquid rosin is the combustible constituent of the explosive.

## *Film-Forming Resins*

**(1) Bent, Franklin A. The production and uses of tall oil ester resins. *Loc. cit.*, Adhesives (9).**

## *Flameproofing Agent*

**(1) Blitz, K., and Simon, W. Addition of fire-retarding agents to high oxidation oils. Monatsschrift fur Textilindustrie 56: 195–198 (1941).**

A study was made of the effect of addition of 1- and 2-naphthol and pyrocatechol on the flammability of tall oil fatty acids. Pyrocatechol was found to have the greatest effect, and 2-naphthol the least.

## *Floor Covering*

**(1) Deutsche Lineoleumwerke A.-G. Manufacture of a material from pine oil for floorings and wall coverings. Swidish patent 80,570 (Feb. 8, 1932); German patent 563,547 (Feb. 17, 1931); T. S. 101: 85.**

Tall oil is mixed with a siccative at high temperatures, then oxidized by blowing air through it. Fillers and coloring agents are added, and the resulting product is rolled or pressed into the desired form.

**(2) Bergstrom, Hilding O. V., and Trobeck, K. G. The preparation of flooring materials from tall oil pitch. Swedish patents 107,074 (June 27, 1938); and 111,119 (Jan. 2, 1939).**

The tall oil pitch is spread on a supporting material or is used to impregnate fibrous materials to produce a material suitable for floor coverings. The tall oil pitch may also be mixed with the neutral oils from tall oil acids. In another method oxidized or cracked pitch is mixed with bark powder, cork meal, or wood meal. Fillers and materials to improve the elasticity may be added, and the mixture is pressed into a boardlike mass.

## *Flotation*

**(1) Erz-und Kohle-Flotation G.m.b.H. The flotation of non-sulfidic ores. German patent 578,819 (Dec. 13, 1930); French patent 721,306 (Aug. 13, 1931); C. A. 26: 4022.**

Resinic acids and oils, which are obtained from the sulphate or sulphite pulping of wood, may be used as covering substances in the flotation of ores.

**(2) Hasselstrom, Torsten. Use of tall oil in flotation process. U. S. patent 1,986,816 (Jan: 8, 1935); T. S. 101: 279; C. A. 29: 1050.**

Tall oil is used for concentrating oxidized ores of the alkaline earth metals. An aqueous pulp of the ore is adjusted to a pH of 7.5–9.0 and agitated with tall oil. A frothy mass is formed and the mass is collected.

**(3) Hasselstrom, Torsten. Tall-oil flotation reagent. U. S. patent 1,986,817 (Jan. 8, 1935); T. S. 101: 297; C. A. 29: 1050.**

A claim is made of the use of refined tall oil as a flotation reagent.

**(4) Hodges, Paul. The use of floating soap and its derivatives. *Loc. cit.*, Emulsifying Agents (8).**

**(5) Jayne, Divid W., Jr. Ammoniated tall oil product as a flotation agent. *Loc. cit.*, Ammonium Salt (1).**

**(6) Pollak, Arthur. Utilization of tall oil. *Loc. cit.*, Emulsifying Agent (14).**

**(7) Vogel-Joergensen, Mikael. Flotation process. British patent 478,275 (Jan. 17, 1936); C. A. 32: 4389.**

Either wholly or partly saponified tall oil is added to the pulp in the flotation of materials that contain calcium carbonate. The carbonate is removed with the froth.

**(8) Wallach, James A. Uses of the fatty acids from pine wood. Soap 13, no. 3: 31–33, 73 (1937); C. A. 31: 3313; T. S. 106: 32.**

The composition of crude and distilled tall oil and some uses of distilled tall oil are given. Among the uses mentioned was the mineral flotation field.

**(9) Christmann, Ludwig J., and Erickson, Stephen E. Use of tall oil in the flotation of nonmetallic minerals. U. S. patent 2,300,827 (Nov. 3, 1942); C. A. 37: 2148.**

In the flotation of nonmetallic minerals, a tall oil phosphate ore promoter is used. It is diluted with about 10–25% of an aliphatic water-soluble ketone to give increased promoting power.

**(10) Geiske, Elmer W. Flotation of tungsten ore. U. S. patent 2,373,305 (April 10, 1945).**

The presence of a reagent combination of an ionic promoter from the group consisting of the higher aliphatic fatty acids, tall oil, and the ammonium, potassium, or sodium salts of fatty acids and tall oil; an alkali salt of a dialkyl sulphosuccinate; and a gangue depressant improves the concentration of carbonate-containing tungstate minerals and silicon-bearing gangue during the flotation step.

**(11) Hoag, Edward H. Flotation reagent. U. S. patent 2,371,292 (March 13, 1945).**

Tall oil; lauric acid ester diethylene glycol ammonium sulphate; and an alcohol of the group consisting of hexanols, heptanols, octanols, nonanols, decanols, and dodecanols constitute a flotation reagent for the concentration of barite ores.

**(12) Mortsell, S. Flotation reagents in Sweden. Tek. Tid., Bergsv. 72: 14–16 (1942).**

In the articles the use of a mixture of tall oil and pine oil as a flotation reagent is mentioned.

**(13) Ried, Robert C. Flotation agent. U. S. patent 2,202,601 (May 28, 1940); C. A. 34: 6561.**

The patent relates to oil-in-water emulsions of mixtures of fatty acids and mineral oil and to their use as collecting agents for calcite and other oxide ore minerals. Tall oil, oleic acid, and fish acid are included in the fatty acids; the mineral oils range from kerosene to bunker C oil. An alkali or ammonium hydroxide is used as the emulsifying agent. Details are given of the amounts of ingredients used.

**(14) Smith, Hugh F., and Vilbrandt, Franc C. Use of paper mill wastes in the flotation of manganese ores. Virginia J. Science 2: 204 (1941); C. A. 36: 1878; B.I.P.C. 12: 371.**

Tall oil is a collector of manganese dioxide in the flotation of low-grade manganese ores. When added to tall oil, other reagents act as frothers, gangue dispersors, or depressors.

**(15) Brown, Earl H., and Tartaron, Francis X. Concentration of oxidized iron ore. Canadian patent 428,699–428,701 (July 10, 1945).**

The aqueous pulp of finely divided iron ores, which has a

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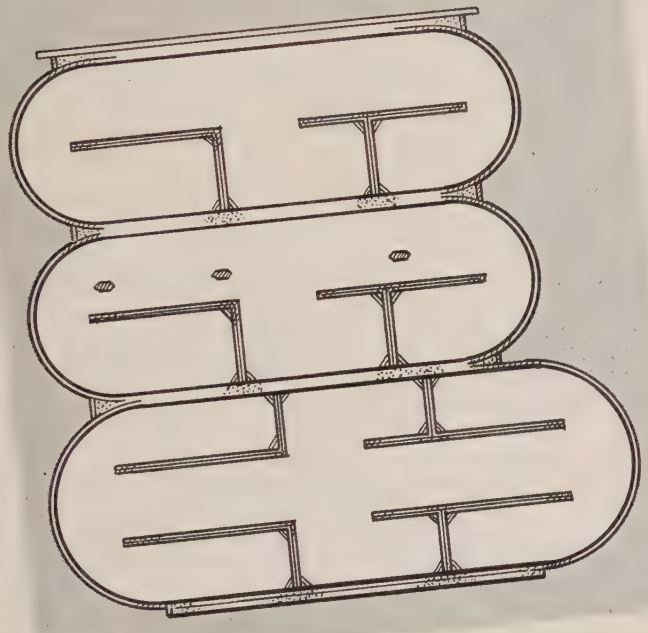

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quartz gangue ingredient, is treated with an anionic collecting agent. The collecting agent is selected from a class consisting of higher fatty and resin acids and their soaps or mixtures of these acids, such as tall oil.

**(16) Christmann, Ludwig J., Jayne, David W., Jr., and Erickson, Stephen E. Flotation of acidic minerals. U. S. patent 2,312,387 (March 2, 1943).**

Promotors for acid minerals in froth flotation, film flotation, agglomeration, tabling, and stratification may be produced by the reaction of polyalkylene polyamines with tall oil. The temperature of reaction is 230–240°C. and the quantities used vary as the resin acid content of the tall oil.

**(17) Clemmer, Julius B., and Clemmons, Ballard H. Flotation of iron ores. U. S. patent 2,383,467 (Aug. 28, 1945); C. A. 39: 5242.**

Tall oil can be used as a collector in the removal of silicia from iron ore. The method of flotation and an analysis of the ore are given.

**(18) Clemmer, Julius B., and Clemmons, Ballard H. Flotation of fluorspar. U. S. patent 2,407,651 (Sept. 17, 1946); C. A. 41: 576.**

Sodium fluoride, a lignosulphonate, and a collecting agent are used to treat a fluorspar pulp. Fatty acid or soap is used as the collecting agent; following this treatment, the mixture is subjected to froth flotation.

**(19) Clemmer, Julius B., and Rampacek, Carl. Concentration of iron ore. U. S. patent 2,403,481 (July 9, 1946); C. A. 40: 5004.**

Tall oil is used as a collector and lime is added to activate the silicates in the flotation of siliceous and calcerous gangue from iron ore by the use of lignosulphonates.

**(20) Clemmer, Julius B., and Rampacek, Carl. Flotation of nonmetallic minerals. U. S. patent 2,424,552 (July 29, 1947); B.I.P.C. 18: 75; C. A. 41: 6182.**

Tall oil may be used as a collecting agent in the beneficiation of ores of nonmetallic minerals, which have been blunged in the presence of hydrated lime to obtain a pH of 11.

**(21) Clemmer, Julius B., and Williams, Milton F., Jr. Flotation of silica from iron ore. U. S. patent 2,419,945 (May 6, 1947); B.I.P.C. 17: 606; C. A. 41: 4764.**

Tall oil or its soaps can be used as the anionic collecting agents in the beneficiation of iron ores by froth flotation of the siliceous gangue constituents from the iron oxides.

**(22) Crawford, Bruce D., and Jayne, David W., Jr. The flotation of phosphate ore. U. S. patent 2,416,909 (March 4, 1947); C. A. 41: 3035.**

Tall oil and coconut oil are the preferred soap and diluent in the froth flotation of phosphate ores.

**(23) Greene, Ernest W., and Head, Charles W. Concentration of phosphate ores. U. S. patent 2,303,931 (Dec. 1, 1942).**

A mahogany soap, a mixture of fatty and resin acids, such as tall oil, and a fuel oil are used to condition the ore in an aqueous pulp in the concentration of phosphate from an ore containing phosphate and silicia particles. By the addition of about 0.5 pound of tall oil per ton of ore, 133–717% more phosphate is recovered in the floated concentrates.

**(24) Herkenhoff, Earl C. Use of sulphonated agents in the magnetic recovery of iron ores. U. S. patent 2,423,314 (July 1, 1947); B.I.P.C. 17: 744; C. A. 41: 5844.**

Sulphonated tall oil can be used as an agglomerating agent for treatment of the aqueous pulp in the concentration of oxidized iron ores from mixtures, which contain acidic ganque and both magnetic and nonmagnetic iron oxides.

**(25) Herkenhoff, Earl C. Froth flotation of silicia from iron ore by the use of anionic collectors. U. S. patent 2,423,022 (June 24, 1947); B.I.P.C. 17: 745; C. A. 41: 5844.**

Various examples are given. In one, the material is subjected to a preheating operation by treating it, at 23% solids, for 1 minute with 0.35 pound of saponified tall oil per ton of ore, then to a ten-minute conditioning period with 2 pounds of hydrated lime per ton of ore. Pine oil in an amount of 0.08 pound per ton of ore is then added as a frother and the ore floated for six minutes.

**(26) Hoag, Edward H. A flotation process. U. S. patent 2,378,552 (June 19, 1945); C. A. 39: 3889.**

A stable flotation reagent; which has strong collecting properties for baryta ores and depressing properties for siliceous materials, limestones, and oxides of aluminum, iron, manganese, and titanium; can be produced from low-cost chemicals. The reagent is composed of 75% tall oil, 20% of the ammonium sulphate derivative of diethylene glycol laurate, and 5% octyl alcohol.

## *Flypaper*

**(1) Schmid, W. Liquid rosin and its uses. Papier-Fabr. 29, no. 1: 1–4 (Jan. 4, 1931); Farben-Chem. 2: 306–310 (1931); Paper-Maker 81, no. 3, suppl.: cvi (March, 1931); T. S. 97: 279; C. A. 25: 3165, 5582; B.I.P.C. 1, no. 6: 12.**

The average yield of liquid rosin from sulphate pulping of wood is 30 kg. per ton of pulp. The rosin may be hydrogenated or distilled and used in the manufacture of sticky flypaper, putty, waterproofing preparations, etc.

## *Foam-Forming Agent*

**(1) Bergstroem, Hilding O. V., and Cederquist, K. N. Preparation of foam for fire fighting purposes. Swedish patent 85,050 (Jan. 22, 1931).**

Use may be made of sulphate soap, after purification or in its original form, as a foam-forming material. Emulsifying properties are improved by the addition of aqueous solutions of metal salts and/or higher alcohols; the addition of phytosterol is also beneficial.

## *Fruit-Tree Spray*

**(1) The use of tall oil. *Loc. cit.*, Emulsifying Agent (17).**

**(2) Burgdorf, K. Use of adhesive, dispersing, and wetting materials for plant protection. Reichsamt Wertschaftsausbau Chem. Ber. PB 52021, 1145–1153 (1942); C. A. 42: 5603.**

This article is a review of used and improved materials. A list of carriers for insecticidal and fungicidal dusts are given. The use of tall oil as an adhesive for spray mixtures is mentioned.

## *Use of Tall Oil in Germany*

**(1) Rooney, John F. Use of tall oil in Germany. FIAT final report No. 1144, June 5, 1947. 22p.**

The report covers the use of tall oil as a substitute for rosin and linseed oil in linoleum and surface coating industries and lists products manufactured by Spangenberg Werke Hamburg-Eidelstandt, Germany. Methods are given for preparing a substitute for linseed oil stand oil, to be used in the paint industry; tall oil Beckosol, a cheap resin used for wire coating and for gasoline tanks; and lumitol, to be used as a linseed oil substitute in enamels and varnishes. Also given are various formulas for linoleum cements, and a gravimetric and volumetric method of analysis of tall oil for fatty acids, rosin acids, and unsaponifiable matter.

## *Hydrogenation*

**(1) Segessemann, Ernest. The treatment of tall oil. U. S. patent 2,276,517 (March 17, 1942).**

One step in a process of treating saponified tall oil, in which the unsaturated fatty portion has been saturated by hydrogenation consists of chilling a solution of the saponified-hydrogenated tall oil in an organic solvent to precipitate the fatty acid soaps.

**(2) Bergstroem, Hilding O. V. Preparation of solid fat-like products. Swedish patent 57,226 (Sept. 3, 1924); C. A. 18: 3730.**

The fraction or fractions containing substances, resulting from the purification of liquid resin by distillation, are treated with hydrogen in the presence of a catalyst at increased temperatures and a suitable pressure.

**(3) Bergstroem, Hilding O. V., and Cederquist, K. N. Hydrogenation of sulphate waste liquor. Iva 1932, no. 2: 40–44; C. A. 26: 3663; T. S. 95: 233.**

Bad odors, resulting from the sulphur content, render sulphate

pulping liquors unsuitable raw materials for by-products. Oils and ether-soluble tars, free from "sulphate odor," have been produced by dry distillation of the liquor with carbon monoxide and hydrogen under pressure and in the presence of suitable catalysts.

**(4) Dittmer, M. Tall oil, by-product of the sulphate pulp process. Z. Angew. Chem. 39: 262–269 (1926); C. A. 20: 2072; B.C.A. 1926B: 414.**

An analysis of tall oil and methods of separating the petroleum-ether-insoluble material and of obtaining hydrocarbon-free phytosterol are given. Stearic acid may be produced by catalytic hydrogenation of the fatty acids and saponification. Previous investigations on tall oil are reviewed in this article.

**(5) I. G. Farbenindustrie A.-G. The transformation of resins into hydrocarbons. German patent 624,843 (April 23, 1926); Chem. Zentr. 1936, II: 2024.**

Tall oil or resins are heated under pressure with a reducing gas containing bound hydrogen at a temperature above their decomposition point.

**(6) Jahrstorfer, Michael, and Schwarte, Georg. Process for preparing a waxlike substance. U. S. patent 2,134,333 (Oct. 25, 1938); C. A. 33: 1068.**

Six hundred grams of tall oil, which have been hardened with hydrogen in the presence of a catalyst of nickel and kieselguhr are heated at 260°C. for four hours with 50 parts of iron powder obtained from the decomposition of iron carbonyl. The hardened tall oil had an acid value of 172, a saponification value of 176, and a melting point of 58°C.; after heating the mixture had an acid value of 35, a saponification value of 46, and a melting point of 76°C. A colorless waxlike product is obtained by mixing with 50 parts of a nickel-kieselguhr catalyst containing 20% nickel and treating with hydrogen under a pressure of 80 atmospheres for five hours at 270°C. The product, which consists mainly of hydrocarbons has an acid value of 0, a saponification value of 1.4, and a melting point of 67°C. It may be used for the preparation of polishing compounds and shoe creams and for dressing and impregnating textile materials.

**(7) Melamid, M. Preparation of fatty acids. British patent 169,962 (Aug. 30, 1921); 170,562 (Sept. 1, 1921); German patent 361,734 (Oct. 3, 1920); 371,525 (Oct. 21, 1920); French patent 539,971 (July 4, 1922); C. A. 16: 848, 1160.**

Tall oil or liquid rosin, produced during the manufacture of sulphate pulp, is hydrogenated under pressure with a nickel catalyst to produce substances of the nature of fatty acids. The products are used in the manufacture of soaps. If the tall oil is purified before hydrogenation, light yellow products are obtained and are useful for oiling textile fibers.

**(8) Riebecksche, Motan-Werke A.-G. A. The preparation of fatty acid-like compounds. German patent 429,272 (Oct. 22, 1921); Chem. Zentr. 1926, II: 1351.**

A liquid product, useful as a substitute for olein in the textile industry, is prepared by treating tall oil with hydrogen until it is odorless.

**(9) Societe Meusienne de produits chimiques. Purification of oils obtained in the manufacture of pulp. French patent 656,728 (June 29, 1928); T. S. 91: 45; 92: 177; C. A. 23: 4343.**

Their bad odor and relatively high content of unsaponifiable matter make oils, obtained as by-products in the manufacture of pulp, unsuitable for use in soap making. However, the oils may be used after washing with cold acid, drying, and hydrogenating in the presence of a catalyst.

**(10) Tetralin G.m.b.H. The preparation of a fat- or soap-like product. German patent 408,512 (Sept. 14, 1922); Chem. Zentr. 1925, I: 1468.**

Liquid rosin is transformed into an ester or a salt then hydrogenated in the presence of a catalyst in order to increase its acid characteristics.

**(11) Colgate-Palmolive-Peet Co. The hydrogenation of unsaturated fatty acid salts. British patent 550,356 (Jan. 5, 1943); C. A. 38: 1657.**

Crude tall oil soaps may be hydrogenated to give light colored material suitable for use in the soap industry. An aqueous solution of the soaps is treated with hydrogen at a pressure greater than 50 pounds per square inch and at a temperature of 40–200°C. A nickel hydrogenation catalyst is used.

**(12) Oliver, Anthony F., and Palmer, Robert C. Isolation of fatty acids from tall oil. U. S. patent 2,280,842 (April 28, 1942); C. A. 36: 5665; B.C.P.A. 1943B, II: 289.**

Hard fatty acids and abietic acid may be isolated from tall oil by hydrogenation then fractional crystallization. Treatment, prior to hydrogenation; with absorbent clay, activated charcoal, or fuller's earth, a heat treatment at a temperature above 250°C. but below the decomposition temperature, and a treatment with a heavy metal compound; remove oxidized bodies and sulphur compounds, which interfere witth hydrogenation methods. A mixture of suboxides of copper and nickel is used as the catalyst during hydrogenation, which is carried out in the liquid phase using pressure as high as 300 pounds per square inch and minimum temperature of 150–160°C.

**(13) Papps, George, and Othmer, Donald F. The separation of stearic and abietic acids of tall oil by selective absorption after hydrogenation. Ind. Eng. Chem. 36, no. 5: 430–434 (May, 1944); C. A. 38: 3181; B.I.P.C. 14: 335; B.C.P.A. 1944B, II: 319.**

Although fractional distillation has been used more for separating the fatty and resin acids of tall oil than any of the proposed methods; it is expensive, yields are poor, and separation is inefficient. In this work, tall oil was hydrogenated to reduce the components to two, and the components were separated by passing a solution through a column containing activated carbon. Individual absorption isotherms were determined and are discussed. Included is a proposed flow sheet for the hydrogenation of tall oil and the separation of the abietic and stearic acids.

**(14) Segessemann, Ernest. Treatment of tall oil. U. S. patent 2,305,498 (Dec. 15, 1942); Canadian patents 424,381 and 424,382 (Dec. 12, 1944); C. A. 37: 3292; B.C.P.A. 1944B, II: 174.**

The patents are concerned with a process of treating hydrogenated tall oil or its esters in order to separate the fatty acids. For example: A product, containing chiefly oleic and resin acids, was obtained by hydrogenating distilled tall oil under a pressure of 10 atmospheres at a temperature of 120°C. for 0.5 hour with 2% nickel. One hundred grams of the product were then dissolved in 800 ml. of acetone and cooled to —60°C. for two hours, giving a yield of 63 grams of oleic acid. Other examples are cited.

**(15) Turck, Joseph A. V. Jr. The preparation of organic materials. U. S. patent 2,309,483 (Jan. 26, 1943); C. A. 37: 3964; B.C.P.A. 1944B, II: 257.**

The invention covers the hydrogenation of unsaponifiable materials, separated from tall oil, to form stable, light colored substances having a pleasant odor. By distilling the hydrogenated material a light aromatic fraction is obtained which may be used as a perfume with bath salts, plastics, rubber, sulphonate derivatives or as a deodorant. Hydrogenation pressures and temperatures are given, and many uses are listed.

**(16) Dressler, Russell G., and Vivian, Robert E. The treatment of and products obtained from tall oil. U. S. patent 2,336,472 (Dec. 14, 1943); C. A. 38: 3152.**

Materials, which act as poisons for hydrogenation catalysts, may be either removed or rendered inert by a pretreatment with natural or activated absorbent clays and earths. Tall oil is not significantly bleached by these materials, and the hydrogenated products have a lighter color, increased stability, are free from stickiness, practically odorless, and physically homogenous. Quick mixing of tall oil, catalyst, and hydrogen gives best results in hydrogenating tall oils. Hot tall oil or its hydrogenation product should be kept in an inert atmosphere of carbon dioxide, hydrogen, or nitrogen. The products may be used for the manufacture of greases, lubricants, rubber, soaps; and to replace commercial oleic and stearic acids, vegetable and animal oils and fats in soaps and textile dressings.

**(17) Dressler, Russell G., and Vivian, Robert E. The hydrogenation of tall oil. U. S. patent 2,369,466 (Feb. 13, 1945).**

Prior to hydrogenation crude oil is mixed with finely divided solid absorbent material at 105–120°C. The tall oil, after removal of the absorbent material, is then heated to a temperature of 150–180°C. in the presence of hydrogen and a catalyst at a pressure of 200–600 pounds per square inch. After the rate of hydrogenation has become low, spent catalyst is removed and fresh catalyst is added. Hydrogenation is continued until the product has an iodine number less than 145, a thiocyanogen number less than 65, and a melting point greater than 25°C.

**(18) Dressler, Russell G., and Vivian, Robert E., and Hasselstrom, Torsten. The treatment of tall oil. U. S. patent 2,358,004 (Sept. 12, 1944); C. A. 39: 634.**

A product, obtained by the pretreatment of tall oil in the presence of a catalyst capable of stabilizing the resin constituents, is subjected to esterification, and separation in the presence of selected solvents. The product, containing esters of tall oil fatty acids, is hydrogenated to obtain a stable product of greater benefited color and odor. Examples are given. A yield of 600 grams of methyl esters of fatty acids, 750–825 grams of resin acids, and 150 grams of unsaponifiable matter was obtained from 1500 grams of tall oil. Hydrogenated fatty acid esters may be hydrolyzed to the free acids or can be used as a substitute of methyl or ethyl oleate.

**(19) Dressler, Russell G., Vivian, Robert E., and Hasselstrom, Torsten. Stable compounds from tall oil. U. S. patent 2,371,230 (March 13, 1945).**

Tall oil, in the presence of a disproportionation catalyst, is heated to 100–240°C. until at least 1% of the resin acids of the formula $C_{20}H_{28}O_2$ is produced. After removal of the catalyst, the treated tall oil is hydrogenated under heat and pressure. A stable compound is obtained and contains resin and fatty acids in practically the same proportions occurring in the original material.

**(20) Harwood, James, and Binkerd, Evan F. Treatment of tall oil. U. S. patent 2,423,236 (July 1, 1947); B.I.P.C. 17: 676; C. A. 41: 6047.**

The fatty acids of tall oil are easily hydrogenated after a pretreatment with an alkaline reagent, such as sodium potassium, calcium hydroxide, sodium or potassium carbonate. From 1–2% of the alkali can be added before or after distillation of the acids. Examples are given.

## *Impregnating Agent*

**(1) Engstroem, J. G. A. An impregnating mass. Swedish patent 47,161 (May 29, 1918).**

The impregnating mass is composed of 53 parts of rapeseed oil, 25 parts of liquid rosin, and 22 parts of sulphur.

**(2) The use of waste products. *Loc cit.*, Emulsifying Agent (18).**

**(3) Dorken A.-G., Ewald. Preparation of impregnating agents from tall oil. German patent 722,247 (July 31, 1937).**

Impregnating agents are prepared by blowing crude tall oil for 35–40 hours at 80–200°C., heating 8–10 hours at 250–300°C., then esterifying. The oil is esterified, after the addition of polybasic alcohols or their anhydrides, in the usual manner with polyvalent alcohols; or phenols or saturated with lime and siccatives and treated with organic solvents.

**(4) Peterson, M., and Son Aktieselskab. Adhesive and impregnating agents for paper and board. *Loc. cit.*, Adhesives (8).**

**(5) Bergstrom, Hilding, O. V., Heijmer, Gustaf B., and Trobeck, Karl G. An oil-hardened fiberboard. *Loc. cit.*, Board (3).**

## *Insecticide*

**(1) Sutter, Josef. The eradication of plant parasites. Australian patent 157,823 (Jan. 25, 1940); C. A. 36:2078.**

A solution of tall oil to which may be added nicotine, tobacco extract, and petroleum, lime or a similar stabilizer is used to combat insect and other plant parasites.

## *Insulating Material*

**(1) Huebschner, Julius. Swedish liquid rosin. Seifensieder-Ztg. 48: 231–232 (1912); C. A. 15: 2177.**

Descriptions of tall oil, tall oil distillate, and tall oil pitch are given. Tall oil pitch; a glassy, brittle mass with a melting point of 85°C., an acid number of 50.3, and a saponification number of 75.5; is used for insulation and cheap grades of black varnish.

**(2) Lagerqvist, J., and Sandstrom, G. Insulating oil. Svensk Kem. Tid. 55: 238–244 (1943); Svensk Papperstidn 47, no. 3: 59–62 (Feb. 15, 1944); B.I.P.C. 15: 76; C. A. 38: 6426.**

Resins, obtained by the distillation of tall oil, may be converted into products fairly soluble in mineral oils with a viscosity between the values of a semiliquid oil and those of easily fusible transformer oils by suitable heat treatment. The dielectric properties of the products given in tables and graphs indicate that the insulating oils thus produced are superior to those prepared from a rosin base and imported from America.

## *Binders for Iron Ore Slimes*

**(1) Klinefelter, T. A. Binders for use in pelletizing slimes. U.S. Bur. Mines, Rept. Investigations 3846. 1946. 13p. C. A. 40: 7111.**

It was found from a study of commercial binders for iron ore slimes and table concentrates that slaking can be prevented by the use of tall oil.

## *Leather Oils*

**(1) Keghel, M. de. The composition of tall oil and its utilization. *Loc. cit.*, Axle Grease (1).**

## *Lime Treatment*

**(1) Bernardi, Dominic J., and Florence, Robert T. Printing ink. U. S. patent 2,416,676 (March 4, 1947); B.I.P.C. 17: 463; C. A. 41: 3639.**

Satisfactory vehicles for inks may be produced by reacting tall oil with lime and preferably mixed with a small percentage of linseed or other drying oil. Tall oil is heated to 180–200°C. and treated with 5% of hydrated lime as the temperature is increased to 220°C. The oil is held at this temperature for 5–6 hours. The effect of resin content upon the viscosity is shown, and formulations are given.

## *Lubricating Oil*

**(1) Heijne, O. H. Lubricating oil from wood tar. Swedish patent 47,004 (Oct. 12, 1917).**

Liquid rosin is used as one of the components of lubricating oil.

**(2) Keghel, M. de. The composition of tall oil and its utilization. *Loc. cit.*, Axle Grease (1).**

**(3) Kleine, C. W. A. Lubricating and boring oils. *Loc. cit.*, Cutting Oils (2).**

**(4) Lieber, Eugene. Lubricants. U. S. patent 2,262,809 (Nov. 18, 1941); C. A. 36: 1772.**

A waxy lubricating oil and a condensation product of halogenated tall oil and a cyclic compound are used to prepare a lubricant.

**(5) Lieber, Eugene. Method for the preparation of a lubricant. U. S. patent 2,270,319 (Jan. 20, 1942).**

The lubricant is essentially composed of a waxy lubricating oil and not more than 5% tall oil.

**(6) Tharens, F. I. A substitute for machine oil. Swedish patent 44,515 (Dec. 1, 1917).**

One of the components of a material to be used as a substitute for machine oil is liquid rosin.

**(7) Harris, Elmer, F. A cold rolling solution. U. S. patent 2,258,552 (Oct. 7, 1941).**

A lubricant, to be used for a material being subjected to rolling, is composed of an emulsion of tall oil, palm oil, triethanolamine, and water.

**(8) Abramowitz, William L., and Beach, Ralph, M. Cutting oils from sulphurization of oils and fats. *Loc. cit.*, Cutting Oils (6).**

**(9) Alsmark, Kurt O. Oil-in-water type emulsions. *Loc. cit.*, Emulsifying Agent (20).**

**(10) Alsmark, Kurt, O. The preparation of lubricants. *Loc. cit.*, Emulsifying Agent (21).**

**(11) Farber, Edward. The utilization of wood acids. U. S. patent 2,367,462 (Jan. 16, 1945).**

Tall oil is mixed with a neutralizing agent, from the group consisting of the inorganic oxides and hydroxides, at a temperature above 100°. The quantity of neutralizing agent should be not more than 33% nor less than 5% of the quantity needed to neutralize the free acidity of the oil. The mass

is heated to about 300°, and evaporated at atmospheric pressure until the residue is neutral. Use is made of the product as a lubricant or to rustproof metals.

**(12) Roveri, Luigi. Preparation of a synthetic oil. Italian patent 392,628 (Oct. 8, 1941); Chem. Zentr. 1944, I: 844.**

A transparent oil, which may be used as a lubricant, is prepared by heating to boiling for 20–30 minutes a mixture of 5–12% of rosin, 4–14% of tall oil, 4–10% of potassium carbonate, and water to make 100%. The product is mixed with 2–7% of xylene and 3–7% of alcohol after cooling.

**(13) Davis, Gifford D., and Barth, Edwin J. Sulphurized glyceryl esters of tall oil. U. S. patent 2,385,912 (Oct. 2, 1945); C. A. 40: 720.**

Tall oil, which has been esterified with glycerol, is sulphurized with sulphuryl halides or oxyhalides, active or elemental sulphur. After sulphurization, the mass is blown with air to remove the disagreeable smelling volatile products. The esters have desirable lubricating properties such as oiliness, tenacity, wetting power, and pressure and anticorrosive properties. They also impart lubricating properties of paraffinic and naphthenic lubricating oils, when dissolved in them.

**(14) Jolly, Samuel E. Lubricants from tall oil. U. S. patent 2,380,531 (July 31, 1945); C. A. 40: 720.**

Tall oil is converted into nitriles by feeding it into the top of a contact column, packed with a dehydration catalyst, through which ammonia is passed countercurrent to the flow of the oil. The oil is reacted with the ammonia to form ammonium salts, and the salts are converted into the amides then to the nitriles. The nitriles are then sulphurized at 190–200° to 20–25% sulphur. Mineral oils, which contain sulphurized nitriles, are nonemulsifiable; they may be used as mild extreme-pressure lubricants, and emulsified cutting oils, and greases. They are noncorrosive to brass, bronze, copper, and aluminum.

**(15) Enkvist, Terje, Makela, Keijo, and Krohn, Tovio. A lubricating oil from pine tar and tall oil. Finnish Paper Timber J. 29, 336–338, 353–356 (1947); C. A. 42: 3943.**

Studies were made on the preparation of lubricating oil from pine tar. The proposed method of preparation is outlined, and methods of extraction or separation of the different components or fractions are discussed. A crystalline fraction, obtained by the crystallization of the petroleum ether-soluble resin acids from 92% methyl alcohol, was similar to the fractions obtained from rosin and tall oil. The unsaponifiable matter, which is pre-existing in the original tar, is useful as a lubricating grease.

**(16) McCleary, Rush F. Lubricating-oil additives for heavy-duty oils. U. S. patent 2,440,375 (April 27, 1948); C. A. 42: 7031.**

Oil-soluble or oil-miscible metal derivatives of alkyl-substituted aryloxy or arylmercapto tertiary amines have proved to be successful as antioxidant, detergent, and anticorrosive agents for use in heavy lubricating oils. The method of preparation of such a compound is described. The patent also includes auxiliary antioxidants to be used in heavy-duty Diesel and gasoline engines. The use of sulphurized tall oil, as a sulphurized anticorrosion compound to be used for this purpose, is mentioned.

**(17) Davis, Gifford D., and Barth, Edwin J. A noncorrosive lubricating composition. U. S. patent 2,431,737 (Dec. 2, 1947); C. A. 42: 1731.**

High pressure and metal-working lubricants are prepared from sulphurized glyceryl esters of tall oil and mineral oil. Sulphurized glyceryl esters of tall oil are prepared by heating 5–17 parts of sulphur with 95–83 parts of glyceryl esters of tall oil at temperatures of 320–350°F. for 2–4 hours. The mixture is then cooled to 200°F. and blown with air for one hour to remove malodorous and unstable produces. The blended lubricant, consisting of 20 parts of sulphurized glyceryl esters of tall oil and 80 parts of mineral oil, is used for cutting, milling, and sawing; it is noncorrosive to metallic surfaces at ordinary temperatures.

## *Maleic Acid Adduct, Esterification*

**See Bulletin No. 7, Bibliography on Paints, Varnishes, Lacquers, etc.**

## *Metal Cleanser*

**(1) Hodges, Paul. A metal cleaning composition. U. S. patent 1,993,097 (March 5, 1935); C. A. 29: 2632; T. S. 101: 297.**

Floating soap is an effective agent for the removal of acid, corrosion, grease, paint, rust, and scale from metal surfaces. The soap alone has remarkable penetrating powers, but is more effective in emulsions with amylene dichlorides, ethylene dichloride, trichloroethylene, diacetone, dioxane, acetone, ethyl lactate, or gasoline. Examples are given.

**(2) Keghel, M. de. The composition of tall oil and its utilization. *Loc. cit.*, Axl Grease (1).**

## *Metallic Salts*

**(1) Burrell, Harry. Process of making tall oil metal compounds. U. S. patent 2,306,352 (Dec. 22, 1942); C. A. 37: 3286; B.C.P.A. 1944B, II: 209.**

Tall oil, when reacted at elevated temperatures with metallic compounds, may be converted into plastic masses capable of hardening by heating. The use of compounds of heavy metals or alkaline earth metals is preferred. Any metal, except those of group one of the periodic table, may be used; and carbonates, chlorides, hydroxides, or oxides of the metals may be used. Examples are given of a number of combinations.

**(2) Elliott, Stanley B. Preparation of metallic soaps. Soap & Sanitary Chem. 18, no. 7: 25–27, 73–74 (July, 1942); Enamelist 19, no. 9: 30–36 (1942).**

Tall oil and a number of saturated and unsaturated fats and fatty acids were used to prepare metallic soaps. For the saponification, metallic salts that are easily soluble in water should be used. Because of the information of soluble alkali soaps, the presence of alkali impurities should be avoided.

**(3) Frick, O. F. V. Sulphate soap which is insoluble in water and stable in air. Norwegian patent 67,360 (Sept. 2, 1941).**

Tall oil acids, sulphate soap, or tall oil pitch is treated with metals, whose hydrates are amphoteric in nature, to prepare a soap insoluble in water and stable in air. Aluminum, iron, or zinc may be employed in the form of oxides, hydroxides, or their easily soluble salts.

**(4) Hastings, R. Use of tall oil for the textile industry. Am. Dyestuff Reptr. 33, no. 2: 25–26, 50 (Jan. 17, 1944); B.I.P.C. 14: 228.**

A number of applications of tall oil to the textile industry are listed, and a brief review of its composition is given. The metal soaps of tall oil find use in the mildewproofing and waterproofing of textile fabrics.

**(5) Hastings, R. Use of tall oil in the paint industry. Am. paint J. 28, no. 3. 42, 46, 48, 50 (1943); C. A. 38: 267; B. C. P. A. 1944B, II: 77.**

Applications of tall oil in the paint industry are given, and its physical and chemical properties are tabulated. The metallic soaps of tall oil are employed as paint driers and wetting agents.

**(6) Long, Kenneth E. Basic lead naphthenate. U. S. patent 2,157,766 (May 9, 1939). Preparation of basic metal salts. U. S. patent 2,157,767 (May 9, 1939); C. A. 33: 6625 and 33: 6626.**

Products, that do not turn yellow, and dry quickly because of high metal content, are prepared by heating fatty acids from tall oil and rosin acids in a high boiling solvent with a metallic oxide or hydroxide of cobalt, lead, manganese, or zinc. Mineral oil, naphtha, or kerosene are the solvents employed.

**(7) Stresen-Reuter, Frederick A., and Rimpila, Charles. Preparation of a water-in-soluble metal soap. U. S. patent 2,175,489 (Oct. 10, 1939); C. A. 34: 899.**

A water-insolube soap of a product derived from pinewood composed of a major portion of unsaturated acids, a minor proportion of rosin acids, and a small proportion of unsaponifiable matter is described.

**(8) Stresen-Reuter, Frederick A., and Rimpila, Charles. Preparation of an alkaline earth metal soap. U. S. patent 2,175,490 (Oct. 10, 1939); C. A. 34: 899.**

A water-insoluble soap comprising the reaction product of an alkali metal compound and a mixture of unsaturated fatty acids, rosin acids, and a small amount of a sterol is described.

**(9) Stresen-Reuter, Federick A., and Rimpila, Charles. Preparation of a water-in soluble soap. U. S. patent 2,175,491 (Oct. 10, 1939); C. A. 34: 899.**

A water-insoluble soap comprises the reaction product of a metal compound and a mixture of unsaturated fatty acids, rosin acids, and a small amount of a sterol and the reaction product of a metal compound.

## *Molded Products*

**(1) Winson, Th. M. Artificial products. German patent 538,799 (May 30, 1929); Chem. Zentr. 1932, I: 2792.**

A molding compound may be made by mixing with the rosin precipitated from black liquor a sizing material a phenol resin, and fibers.

**(2) Bergstrom, Hilding O. V., and Trobeck, K. G. Pressed masses from vegetable materials and salts of fatty and rosin acids. Swish patent 104,606 (Aug. 1, 1938).**

An acid-reacting salt, such as alum or sodium acid sulphate is used to impregnate a vegetable material. The impregnated material is then treated with fatty or resin acids or their salts and the material dried and pressed hot. It may be hardened by the action of carbon dioxide, sulphur dioxide or similar gases at a temperature of 150–200°C.

**(3) Herbig-Haar haus A.-G. Lackfabrik. Resinous condensation products. German patent 679,046 (Dec. 1, 1934).**

The ester, resulting from the esterification of one mole of erythritol with 2 moles of tall oil distillate until the acid number is about 2, is then esterified with an ester from 2 moles of methyladipic acid and 2 moles of glycerol at 220°C. The resin may be used in the preparation of pressed materials and as an impregnating agent.

## *Oil Well Drilling*

**(1) Anderson, Francis M. An oil-base drilling fluid. U. S. patent 2,430,039 (Nov. 4, 1947); C. A. 42: 1047.**

A formula for an oil-base drilling fluid is: Diesel oil and air-blown asphalt blend 38,750 gallons, sodium silicate 13,200 pounds, tall oil 13,200 pounds, sodium hydroxide 900 pounds, and saturated sodium chloride in water 1050 pounds. The drilling fluid is suitable for drilling through anhydrite formations.

**(2) Self, Edward S. An oil-base drilling fluid. Canadian patent 441,616 (May 20, 1947); C. A. 41: 5294.**

An oil-base drilling fluid is composed of a mineral oil, a finely divided suspended solid, and a stabilizing agent. Formulas are given.

## *Paint Remover*

**(1) Hodges, Paul. A paint, varnish, and lacquer remover. U. S. patent 1,993,096 (March 5, 1935); T. S. 101: 297.**

Floating soap may be used with amylene dichlorides, alcohol, oleic acid, or water to prepare the emulsion. Other solvents may be used, and tall oil may be substituted for the floating soap. One example is as follows: floating soap 10%, oleic acid 3.3%, amylene dichlorides 20%, and water 66.7%.

## *Pentaerythritol Esters*

**See Bulletin No. 7, Bibliography on Tall Oil in the Paint, Varnish, Lacquer, Core Oil, Ink, and Related Industries.**

## *Petroleum Industry*

**(1) Dittler, K. Tall oil in the alkali wash of petroleum distillates. Chem.-Ztg. 52: 577–578 (1928); C. A. 22: 4238; T. S. 89: 80.**

The analysis of a typical tall oil is given. Tall oil is a good substitute for oleic acid to prevent formation of emulsions during the alkali washing of petroleum distillates. The product is heated with indirect steam to a temperature of at least 85°C., and, after settling for 12 hours, the clear solution is drawn off. 3–5 Baumé sodium hydroxide gives best results. Oils refined with this solution are clear and stable, but may have a brown color.

## *Pharmaceutical Preparations*

**(1) Schmatalla, O. Liquor Cresoli Saponatus. Pharm. Zentralhalle 68: 623–624 (1927).**

The linseed oil, which is used in the preparation of this liquor, may be replaced by tall oil.

## *Plastic Masses*

**(1) Melamid, M. An oil for foundry use. German patent 386,062 (Nov. 18, 1921); Chem. Zentr. 1924, I: 836.**

A plastic mass is formed by treating the distillation residue of fats, oils, or resins with sulphur. The plastic mass may be used in the hot condition or in solution in a suitable light or medium oil.

**(2) Melamid, M. The preparation of plastic masses. German patent 434,143 (Feb. 7, 1923); Chem. Zentr. 1927, I: 678.**

Materials, which may be used as plastic masses for insulation and packing, are prepared from the product obtained from the treatment of the distillation residues of fats, resin-like compounds, and tar-oil distillates with sulphur mixed with asbestos, or with acetylene condensation products, with or without indifferent products. These products may be molded under heat and pressure.

**(3) Obst, W. Utilization of tall oil. *Loc. cit.*, Cutting Oils (3).**

**(4) Sackett, Ernest D. A plastic composition for shoe fillers. U. S. patent 2,121,745 (June 21, 1938); C. A. 32: 6358.**

A plastic composition is prepared from the distillation residue obtained from a substantially neutral, water-insoluble fraction, such as liquid rosin. A batch of extractive material was made by the distillation of acidified soda pulp liquor to a melting point of 140–145°F. It had a penetration of 4.1–7.9 mm. by a No. 2 penetrometer needle under 50 grams pressure for five seconds at 77°F. A mixture was made using 68 grams of this residue, 1.5 grams of calcium hydroxide, 5 grams of rosin, and 21–23 grams of 20–30 mesh comminuted cork.

**(5) Sinit A.-G. The preparation of plastic masses. German patent 455,551 (Dec. 5, 1923); Chem. Zentr. 1928, I: 1600.**

For the preparation of plastic masses, distillation residues of natural resins, tall oil, etc., are heated with sulphur and mixed with indifferent materials, such as asbestos.

**(6) Burrell, Harry. Process of making tall oil metal compounds. *Loc. cit.*, Metallic Salts (1).**

**(7) Daimler, Karl. Resinous masses which contain sulfur and chlorine. German patent 721,792 (May 7, 1942); C. A. 37: 4832.**

Tall oil distillation residues, sulphate pitch may be treated with 10–20% of sulphur halide at 60–70°C. to produce resinous masses.

**(8) Farber, Eduard. Resinous products for oilproof varnishes. U. S. patent 2,312,807 (March 2, 1943); C. A. 37: 4832; B.C.P.A. 1944B, II: 331.**

A mixture of one of the resin and fatty acids of tall oil and a cellulosic substance is heated to about 300°C. to produce a resinous material for use on ceramic materials and metals. The weight of acid used should be at least as great as that of the cellulosic substance.

**(9) Farben, Eduard. Resinous products. British patent 546,384 (July 10, 1942); C. A. 37: 4162.**

A resin is produced by heating polymerized carbohydrates with high-boiling wood acids, such as those contained in tall oil or wood resins. Because of the absence of reaction-inducing substances, the resin tends to have a higher dielectric strength and a higher electrical resistance than previously known resins.

**(10) Wolff, Werner. Plastic masses. U. S. patent 2,298,-078 (Oct. 6, 1942); French patent 858,464 (July 28, 1939); German patent 728,038 (Oct. 15, 1942); C. A. 37: 1530, 6773.**

Polymerized tall oil may be esterified with saturated alcohols in a mixture with vinyl ethers, which have a plurality of vinyl groups in the molecule, to obtain interpolymerization products capable of wide use. Glycol and polyglycoldivinyl ethers or octadecanedil divinyl ethers, vinyl ethers of mono-

hydric alcohols and vinyl esters, acrylic compounds, and other vinyl compounds may be used. During the polymerization fillers, indifferent resins, pigments, waxes, and similar substances many be present. The product may be prepared by the addition at about 10°C. of 5% solution of boron fluoride dihydrate in dioxane (in an amount that noticeable polymerization takes place) to a mixture of 12.7 parts of tall oil glycerol ester, 4.7 parts of diethylene glycol divinyl ether, 28 parts of vinyl ethyl ether, and 8.7 parts of rosin and maleic anhydride and subsequent esterification with glycerol. The finished product; a brown, friable, elastic mass similar to linoxyn when blown with air or oxygen becomes more plastic allowing more fillers to be added.

## *Putty*

**(1) Harkko, E. The preparation of putty. Swedish patent 45,534 (Dec. 3, 1915); British patent 15,248 (Oct. 28, 1915); C. A. 11: 1893.**

A putty may be prepared by boiling 700 parts of rosin oil until frothing ceases then adding, with stirring, 300 parts of petroleum. To this mixture is added chalk.

**(2) Harko, E. Preparation of putty. British patent 15,249 (Oct. 28, 1915); C. A. 11: 1893.**

A putty is prepared by adding chalk or whiting to a mixture of glue, petroleum and bolted meal, rosin oil, and water. 50 parts each of petroleum and bolted meal, 350 parts of rosin oil, and 500 parts of water are used.

**(3) Schmid, W. Liquid rosin and its uses. *Loc. cit.*, Flypaper (1).**

**(4) Strandel, Bjorn. The manufacture of putty. Finnish patent 12,871 (May 4, 1928); T. S. 92: 280.**

Floating rosin, the waste oil from the manufacture of sulphate pulp, is used in place of linseed oil, with a large or small amount of vegetable or animal oil, in a process for the manufacture of putty.

## *Rayon*

**(1) Phrix-Arbeitsgemeinschaft. Improvement of the spinnability of cellulose hydrate fibers. German patent 742,523 (Oct. 21, 1943).**

The spinnability of cellulose hydrate fibers is improved by the application of an emulsion of the fatty acid conponents of tall oil or of their esterification product to the fibers. Emulsification is carried out with the aid of an emulsifier.

## *Rayon Desizing*

**(1) Kollmann, Leo. Tall oil in textile processing. *Loc. cit.*, Cotton Bucking (1).**

## *Review*

**(1) Frydelender, J. H. Uses of solid and liquid resins. Rev. prod. Chem. 37: 33–36, 65–70 (1934).**

A description is given of the composition, analysis, properties, preparation, and uses of the liquid and solid resins of tall oil.

**(2) Sandquist, Hakan. Om Tallolja Ing.-Fiss. Akad. Stockholm, Abh. No. 10. 1922. 69 p.**

A general survey of the history, manufacture, and properties of tall oil is given.

**(3) Wise, Louis E. Tall Oil. Paper Ind. 24, no. 8: 822–826, 831 (Nov., 1942).**

A general review, with 47 references, of the purification and uses of tall oil.

**(4) Hess, M. The uses of tall oil in the paint industry. Paint Technology 11, no. 128: 299–304 (Aug., 1946); C. A. 41: 3305.**

A study was made of the terminology, the methods of refining, neutralization, and separation of the fatty and rosin acids. It was found that the drying properties of tall oil media are not always satisfactory, unless they are with oils such as tung.

**(5) Juvonen, V. V. Tall oil. Suomen Kemistilehti 20A, no. 2: 18–28 (1947).**

Included in the review are 48 references.

**(6) Hermann, F. J. Tall oil review. Centrall Inst. Materiaal Onderzoek, Afdel. Verf., Circ. 44, 8 pp. (1948); C. A. 42: 5219.**

This article is a review of the constitution, fractionation, isolation, refining, and chemical changes of tall oil with respect to its uses in the paint industry.

**(7) Frylender, J. H. The uses of tall oil or liquid resin. Rev. prod. chim. 50, 6–9, 23–27 (1947); Chem. Zentr. 1947, I, 477; C. A. 42: 9170.**

This is a review of the papers and patents on the manufacture, composition, extraction, properties, and uses of tall oil and tall oil acids.

**(8) Wheller, P. H. Tall oil. Central Mfg. District Mag. 32, no. 9, 33–37 (1948); C. A. 42: 9176.**

In the article is given a brief description of the production, composition, and common uses of tall oil.

## *Road Building*

**(1) Schmid, W. Liquid rosin and its uses. *Loc. cit.*, Flypaper (1).**

## *Rosin Oil Substitute*

**(1) The use of waste products. *Loc. cit.*, Emulsifying Agent (18).**

## *Rotproofing Agent*

**(1) Marsh, Paul B., Greathouse, Glenn A., Bollenbacher, Katharina, and Butler, Mary L. Use of copper soaps as rot-proofing agents on fabrics. Ind. Eng. Chem. 36, no. 2: 176–181 (Feb., 1944); C. A. 38: 1121.**

Tests were conducted to evaluate copper tallate as a rot-proofing agent. It is highly insoluble in water; it may be solubilized by acid hydrolysis or by reaction with materials which form soluble copper complexes. Fabrics, which have been treated with the soap, lose copper at points where the fabrics are in contact with the soil.

## *Rubber*

**(1) Tengler, J. Rubber-containing varnish and adhesive. *Loc. cit.*, Adhesives (4).**

**(2) La Crosse, Clifford G. Rubberlike compounding material. U. S. patent 2,421,627 (June 3, 1947); B.I.P.C. 17: 676; C. A. 41: 5336.**

Equal parts of tall oil and No. 480 styrene-type resin; which is a product containing polymers of styrene with some polymers of the isomeric methylstyrenes, coumarone, and indene; are used to form a compounding material for GR-S (Buna S) synthetic rubber. The proportions may be varied, but the melting point is lowered when more than two parts of tall oil and one part of styrene are used. About 20 parts of this material may be used with 100 parts of rubber.

**(3) Harber, W. I., and Yoran, C. S. Use of tall oil esters as plasticizers for GR-S. Ind. Eng. Chem. 37, no. 10: 953–956 (Oct., 1945); B.I.P.C. 16: 114; C. A. 39: 5541.**

Esters of tall oil were tested, and the results were compared with those obtained with commercial plasticizers. The tall oil esters tested were: allyl, benzyl, bornyl, B-phenylethyl, cinnamyl, cresyl, cyclohexyl, diethylene glycol, ethylene glycol, glycerol, isopropyl, methylphenylcarbinyl, methyl, nonaethylene glycol, *p*-chlorophenyl, pentaerythritol, phenyl, *p-tetr*-amylphenyl, tetraethylene glycol, thymyl, 2-chloro-*4-tert*-amylphenyl, tetrahydrofurfuryl, 2-chloro-*4-tert*-amylphenyl, and 2-napthyl. Using aromatic-aliphatic alcohols produced marked changes in the vulcanizates, and the time required to incorporate the esters was lowest of any classes studied. Various properties are discussed.

**(4) Pollak, Arthur. Utilization of tall oil. *Loc. cit.*, Emulsifying Agent (14).**

## *Rust Inhibitor*

**(1) Hodges, Paul. A rust-inhibitor. U. S. patent 1,993,099 (March 5, 1935); C. A. 29: 2632; T. S. 101: 297.**

The composition of a rust inhibitor is as follows: 0.5–25 parts of floating soap, about 10 parts of alcohol, and water to make a total of 100 parts. Articles, which are to be treated or freed of grease, may be treated with the solution by either dipping or spraying.

(2) **Farber, Eduard. The utilization of wood acids.** ***Loc. cit.*****, Lubricating Oil (11).**

(3) **Kollen, Myron H., and Flaxman, Marcellus T. A rust-preventive composition. U. S. patent 2,418,075 (March 25, 1947); C. A. 41: 3739.**

Formulas are given for a number of antirust compositions. One such composition contain 5–95% oil-soluble polyvalent metal tall oil soap and 5–85% oil-soluble polyvalent metal degras soap.

## *Silk Degumming*

(1) **Kollmann, Leo. Tall oil in textile processing.** ***Loc. cit.*****, Cotton Bucking (1).**

## *Sizing Agent*

(1) **Baird, P. K., and Curran, C. E. A statistical survey of rosin as used in the paper industry. Paper Trade J. 111, no. 1: 37–44 (July 4, 1940); Tech. Assoc. Papers 23, no. 1: 262–270 (June, 1940); Paper Making and the Printer 59, no. 3: 18–20 (Autumn, 1940); B.I.P.C. 10: 463.**

Tall oil is used with dark rosin for certain low-grade paper products. Tall oil can hardly be considered as a substitute for rosin, and its combination with rosin probably increases the use of the rosin by improving its plastic properties.

(2) **Bergstroem, Hilding O. V., and Cederquist, K. N. Preparation of a sizing agent from rosin acids. Swedish patent 95,088 (March 1, 1935); Chem. Zentr. 1940, I: 167.**

In the preparation of a sizing agent, the rosin acids are melted with a sufficient amount of resinate to lower the melting point of the mixture below 100°C.

(3) **Christiansen, Magnus. The activities of the Research Institute for Paper Industry (Norway) during the war year. Papir-J. 28, no. 23: 210–213 (Dec. 19, 1940); C. A. 35: 3440; B.I.P.C. 11: 375.**

The value of tall oil as a substitute for rosin in sizing is given in a brief summary. The rosin component gives a good size as papermakers' rosin, but the fatty acid component reduces the value of tall oil as a size.

(4) **Continentale A.-G. fuer Chemie. Sizing paper. German patent 454,005 (Jan. 28, 1926); Zellstoff u. Papier 8: 98.**

An emulsion used for sizing paper is prepared by the saponification of pinabietic acid, obtained from tall oil.

(5) **Hasselstroem, Torsten. Preparation of a rosin for paper sizing from tall oil rosin acids. Finnish patent 11,455 (Nov. 23, 1925); Papier-Fabr. 25: 494.**

Rosin acids from crude or purified tall oil are washed with a solvent and melted with acids, bases, or salts to prepare a rosin suitable for paper sizing and soap manufacture.

(6) **Johnstone, Edward J. A sizing composition for paper. U. S. patent 1,929,115 (Oct 3, 1933); T. S. 98: 246.**

To prepare a sizing composition, add 100 parts of resin to an aqueous solution to the amount of 40 parts (by weight) of the rosin which is dissolved 20 parts (by weight) of soda ash. After this mixture has been cooked until the mass is molten, 10 parts (by weight) of rosin of swedish tall oil are added. Further cooking for about five hours or until homogenity is secured produces a chemical or quasichemical, or colloidal paste. As much as 30 parts of tall oil may be used, and it reduces the melting point of the rosin and decreases the drying time of the paper.

(7) **Keghel, M. de. The composition of tall oil and its utilization.** ***Loc. cit.*****, Axle Grease (1).**

(8) **Klemm, P. Rosin substitute. Chem.-Ztg. 34: 1353 (1910); J. Soc. Chem. Ind. 30: 37.**

Liquid rosin may be used as a partial substitute for colophony.

(9) **Klemm, Paul. The sizing of paper. German patent 310,076 (May 8, 1917); Chem. Zentr. 1919, IV: 690.**

A colloidal solution of montan wax is produced by the addition of less than 25% of saponified tall oil to the wax. Size resistance is increased by the use of tall oil.

(10) **Kylander, Yrio. The by-products of the pulp industry. Finnish Paper and Timber J. 6, no. 7: 182–183; no. 9: 264–265 (April 15, May 15, 1926); Wochbl. Papierfabr. 57, no. 37: 1023–1024 (Sept. 11, 1926).**

The rosin acids, obtained from tall oil by distillation, may be used for sizing brown wrapping papers.

(11) **Liquid rosin from tall oil. Svensk. Pappersmasse-Tidn. 14: 550–552 (1937); Auszuge 1938: 67.**

The average composition of tall oil is given, and a number of its uses are listed. Three fractions; fatty acids, a rosin fraction, and pitch; are obtained by vacuum distillation of tall oil. The rosin fraction, which has a melting point higher than colophony, is suitable for paper sizing.

(12) **Neitzke, Oscar F. Manufacture of sized paper. U. S. patent 2,093,337 (Sept. 14, 1937); C. A. 31: 8198; T. S. 106: 154.**

The liquid resin, separated from spent alkaline pulping liquor, is distilled to obtain a substantially nonvolatile, thermoplastic, pyrogenous residue. This residue, while in a molten condition, is intimately mixed with an aqueous liquid to effect its emulsification. The emulsion is then incorporated with a stock from which the paper is formed.

(13) **Neumann, K.-E. Impressions from Swedish sulfate mills (1935). Wochbl. Papierfabr. 67, no. 7: 125–128 (Feb. 15, 1936); B.I.P.C. 6: 325.**

Almost all Swedish mills recover the raw sulphate soap formed during the cooking process and purify the fatty and resin acids. At the time the resin acids were being used as size in several of the mills, and plans for large-scale production were being made.

(14) **Schacht, F., Komm.-Ges. Sizing for paper and cardboard. German patent 624,133 (Jan. 13, 1936); C. A. 30: 2760.**

The acids, obtained from the distillation of tall oil (under vacuum), are saponified and used either alone or mixed with saponified wax as a size for cardboard or paper.

(15) **TAPPI. Disposal of sulfate black liquor soap. Special Report No. 152. April 10, 1931. 6 p.**

The soap, which collects in black liquor storage tanks, may be burned or chemically processed to recover pine oil and fatty acids. They may be sold or treated for the preparation of sizing material. Data are given on the separation of the soap from the liquor and yields per ton of pulp.

(16) **Winson, Th. M. Artificial products.** ***Loc. cit.*****, Molded Products (1).**

(17) **Zachimmer & Schwarz Chemische Fabrik. Dolan. Paper-sizing. German patent 597,506 (May 26, 1934); C. A. 28: 5669.**

The reduction products from resins or tall oil are used to size paper. The products are alcoholic in character and are used with other sizing agents.

(18) **Porter, Rotheus B., Jr. Aqueous dispersions of thermoplastic hydrocarbons for sizing paper and board stocks. U. S. patent 2,356,882 (Aug. 29, 1944); C. A. 39: 417.**

The liquid resin, separated from spent alkaline pulping liquor, is distilled to obtain a substantially nonvolatile thermoplastic pyrogenous residue. A thermoplastic hydrocarbon material, such as that of asphalt or paraffin, is then melted with about 5–15% of the residue, and the melted mixture is mixed with an aqueous solution containing about 0.25–5% of a caustic alkali and about 0.25–2% of an alkali metal pyrophosphate. The pyrophosphate serves as a dispersion promoter and stabilizer for the dispersion of the hydrocarbon material in the aqueous solution. Numerous details are given.

(19) **Schwalbe, C. G. The preparation of strong and thick paper. German patent 381,144 (Aug. 21, 1918).**

An emulsion of tall oil in a soap solution or a solution of tall oil in an organic solvent is used to size paper. The sized paper is dried at 100–120°C.

(20) **Bent, Franklin A., and Peterson, Elbert A. The production and uses of tall oil ester resins.** ***Loc. cit.*****, Adhesives (9).**

(21) **Enkvist, Terje. Some studies of tall oil pitch. Svensk Papperstidn. 50, no. 16: 363–369 (Aug. 31, 1947); B.I.P.C. 18: 202.**

Samples of tall oil pitch from various mills were compared as to composition. These compositions are given and vary

greatly. From sizing experiments conducted with isolated components of the pitch, it was found that it is the resin acids which form the principal active ingredient in the preparation of size for kraft paper and wallboard. The use of the readily esterifiable acids of the pitch imparts poor sizing strength. 14 references are given.

**(22) Juvonen, V. V. Use of tall oil rosin as a sizing agent in the paper industry. Finnish Paper and Timber J. 28, no. 1: 8–13 (Jan. 15, 1946); B.I.P.C. 16: 370; C. A. 40: 4881.**

Upon distillation of tall oil a rosin fraction is obtained; and when the rosin content of the distilled tall oil fractions is more than 35%, part of the rosin crystallizes but can be removed by centrifuging. The rosin crystals are melted and mixed with 2–3% soda to prevent recrystallization on cooling. Although tall oil rosin is readily soluble in alkalies, its tendency to crystallize and its high melting point, 150–155°C., cause difficulties for the preparation of size. Tall oil rosin is most suitable for the Delthirna sizing process, but can also be converted to size in an ordinary indirect steam-heated kettle. However, the finely crushed rosin must be dissolved in alkali in the latter process. Rosin may be used in a mixture of as much as about 50% in the Bemoid or similar processes, in which only part of the rosin is first melted and saponified. In laboratory and large-scale mill tests the sizing properties of tall oil rosin were equal to those of colophony rosin.

**(23) Juvonen, V. V. Use of tall oil size as substitutes for rosin. Finnish Paper and Timber J. 27, no. 17: 256–257 (Sept. 15, 1945); B.I.P.C. 16: 312; C. A. 40: 2000.**

Tall fatty acids, crystallized tall rosin acids, and tall pitch are the main products obtained from the distillation of raw tall oil. A rosin can be prepared from the crystallized fraction and has good sizing ability. No cooking of the size is required, and it may be dissolved to a solution of desired strength and added to the heater. Best sizing results are obtained with steamed groundwood and unbleached sulphate, but it can be satisfactorily used with ordinary groundwood and unbleached sulphite pulp. Due to dark color it cannot be used for bleached pulp.

**(24) Enkvist, Terge. Investigation of tall oil pitch. Svensk Papperstidn 50, 363–369 (1947); C. A. 42: 1420.**

Tall oil pitch is the residue remaining from the distillation of crude tall oil under vacuum. Four samples of pitch were obtained from different Swedish distillations and analyzed and compared. Tall oil pitch has been extensively used as a sizing agent for kraft paper and wallboard. Sizing tests showed that resin acids that were oxidized, unoxidized, or condensed, produce a sizing effect; but the fatty acid fractions were poor sizing agents.

## *Soap*

**(1) Edwards, Karl B. Wood pulp by-products and the soap industry. Perfumery Essential Oil Rec. 35: 667–668 (1944); Oil & Soap 22: 28.**

This is a description of soaps made from tall oil and a discussion of its future possibilities.

**(2) Hammel, E., Schroder, A. W., and Hamesath, H. Preparation of a soap from fatty acid components of tall oil. Swedish patent 110,745 (May 31, 1940).**

To prepare a soap, tall oil is stirred with water then saponified with concentrated alkali or a mixture of alkali with ammonium hydroxide. Crude tall oil should be purified by centrifuging before saponification.

**(3) Hastings, R., and Pollak, A., and Wafer, J. M. Use of tall oil in soap. Soap Sanit. Chemicals 19, no. 5: 24–27, 70 (1943); C. A. 38: 5424; B.C.P.A. 1943B, II: 289.**

Production of tall oil in the U. S. in 1942 is described, and typical analysis and methods of production of liquid and paste soaps are given. As much as 25% tall oil may be used in chip, hard, and powder soaps.

**(4) Houpt, Alfred G. The preparation of powdered soap. U. S. patent 2,366,334 (Jan. 2, 1945); C. A. 39: 2000.**

In the patent is included a process for the separation of the fatty acid soaps of black liquor soap from the rosin acids. A free-flowing soap powder is obtained by cooling an organic solvent solution to precipitate the soaps of the fatty acids, separating them from the mother liquor, and drying under vacuum at a temperature not greater than 80°C.

**(5) Application of rosin and tall oil in the soap industry. Soap, Perfumery and Essential Oil Record 33: 260–262 (1942).**

**(6) Treffler, Andreas. Pros and cons of tall oil in soap. Soap Sanit. Chemicals 20, no. 6: 29–30, 63 (1944). See Bulletin No. 6, Tall Oil in the Soap and Detergent Industry.**

**(7) Clayton, Benjamin. The refining of animal or vegetable oil with simultaneous production of a soap derived from tall oil and fat acids. U. S. patent 2,390,990 (Dec. 18, 1945); C. A. 40: 3285.**

The oil, which is to be refined, and sodium carbonate or sodium or potassium hydroxide (alkali refining agent) are mixed in controlled proportions and pumped into a mixing device. Phosphatides and gums are precipitated, oil and soap stock are heated to vaporize impurities and water, and the mixture is discharged into a vacuum dehydrator at a temperature of 140–230°F. A mixture of tall oil and the dehydrated oil and soap stock mixture is centrifuged at 100–250°F. The separated refined oil and soap stock are purified; the refined soap stock, a molten, anhydrous soap, is then milled or plodded as a solidified soap.

**(8) Dreger, Emil E. A method of treating tall oil. U. S. patent 2,240,365 (April 29, 1941); C. A. 35: 5536.**

Liquid resin from black liquor is subjected to a regulated heat treatment, at relatively high temperature; and, at the same time, a strong current of steam or inert gas is passed through the resin. The heat treatment may be conducted with or without pretreatment and during or after saponification. Reduced pressure and the absence of air or liquid water are advantageous. The unsaponifiable matter is removed and the characteristics of saponifiable and unsaponifiable material are apparently changed. The product is free of objectionable odor and, when grained, forms a harder soap. The process is illustrated by examples. In one example 2000 parts of crude soap are saponified completely with 35 parts of 50° Be'. aqueous caustic soda solution. The soap solution, after boiling with air blowing for several hours, is then grained out with sodium chloride brine. The solution is salted out several times, and the fatty acids are fractionated at 5 mm. Products, substantially free from unsaponifiable constituents, are obtained by subjecting the fatty acid fractions to the alkali-steam treatment. The oxygenated inorganic acid derivatives of the unsaponifiables, as well as the new soap composition may be used in combination with the common auxiliary agents used in soaps and detergents.

**(9) Bergstrom, H. O. V., Heijmer, G. B., and Trobeck, K. G. Use of tall oil in metal soaps. Swedish patent 120,174 (Nov. 11, 1947); C. A. 42: 7557.**

Fractions of tall oil or tall-oil fatty acids, which contain appreciable amounts of emulsifying substances, are used in the preparation of metal soaps. The final fractions of tall oil and the first fractions of the fatty acids are generally used.

## *Soil Stabilization*

**(1) Miller, Abraham B. The stabilization of soil. U. S. patent 2,357,124 (Aug. 29, 1944).**

An aqueous suspension of tall oil and substantially petroleum hydrocarbon-insoluble pinewood rosin can be used to stabilize soil. The suspension should contain a dispersing agent. Soil stabilization is a treatment which renders the soils more suitable road-building materials. Although tall oil alone produces a slight stabilizing action, it is inferior to a combination of 1 part of tall oil to 2 parts of resin. The mixture is ground to pass a 40-mesh sieve then mixed with an alkaline agent, such as sodium or potassium hydroxide, sodium silicate, or trisodium phosphate. A portion of the tall oil, saponified by the alkali, serves as a dispersing agent. Satisfactory results are obtained by the use of 1–10% of the tall oil-resin mixture, based on the weight of the soil. The suspension may be simply sprinkled over the road in some cases.

## *Stabilization*

**(1) Dressler, Russell G., Vivian, Robert E., and Hasselstrom, Torsten. The treatment of tall oil. *Loc. cit.*, Hydrogenation (18).**

**(2) Dressler, Russell G. Vivian, Robert E., and Hasselstrom, Torsten. Stable compounds from tall oil. *Loc. cit.*, Hydrogenation (19).**

**(3) Hasselstrom, Torsten. The art of stabilizing tall oil materials. U. S. patent 2,311,386 (Feb. 16, 1943); C. A. 37: 4572; B.C.P.A. 1944B, II: 292.**

Tall oil or abietic acid from tall oil is heated with iodine to obtain a material, which has much greater stability than Steele's abietic acid. A product, resembling vitamin D and having a higher melting point and in other ways differing from original tall oil, is produced by the treatment of the sterols of tall oil with iodine. The iodine treatment should be carried out at a temperature of 150–200°C. using 0.5–1% iodine; above 3% iodine active halogenation takes place, and below 0.1% iodine no reaction takes place. After heating tall oil with iodine, on standing the resin acids separate. They largely consist of dehydroabietic and dihydroabietic acids; the mother liquor contains the fatty acids, sterols, unsaponifiables, and some resin acids. Other examples are given.

## *Stencil Ink*

**(1) Davis, Alex B. Inks. U. S. patent 2,385,613 (Sept. 25, 1945); C. A. 39: 5511.**

Tall oil is used as one of the components of a stencil ink. The stencil duplicating ink has as its components: tall oil, carbon black, castor oil, ethylene glycol, and triethanolamine.

## *Reaction With Sulphides*

**(1) Boehme, A.-G., H. Th. Capillary active ester-like sulfuric acid compounds. *Loc. cit.*, Emulsifying Agent (3).**

## *Sulphonated Tall Oil*

**(1) I. G. Farbenindustrie A.-G. The preparation of sulfonic acids from fats and oils. German patent 501,086 (Jan. 27, 1938).**

In the preparation of sulphonic acids, tall oil and other oils and fats are treated with chloroacetylchloride sulphonic acid.

**(2) I. G. Farbenindustrie A.-G. A method for preparing true sulfonic acids from unsaturated aliphatic or hydroaromatic hydrocarbons and their derivatives. German patent 550,243 (June 5, 1927); C. A. 26: 4067.**

True sulphonic acids, which are stable to boiling hydrochloric acid, are obtained by the treatment of unsaturated aliphatic or hydroaromatic hydrocarbons or their derivatives with chlorosulphonic acid or fuming sulphuric acid in the presence of esters or low-molecular weight ethers; or by treatment of the hydrocarbons or their derivatives with esters of chlorosulphonic acid or substances forming these esters. The amount of sulphonating agent must exceed that required to esterify the hydroxyl group, when hydroxylated unsaturated compounds are treated. One hundred parts of tall oil were treated with 160 parts of ethyl chlorosulphonate. The mixture was then heated for two hours at 80°C., and the reaction product diluted with water and heated under reflux with sulphuric acid.

**(3) Nill, Edward A. Method of making a drawing composition. *Loc. cit.*, Drawing Oil (1).**

**(4) Oranienburger chemische Fabrik A.-G. The preparation of high molecular sulfonation products. German patent 583,606 (Aug. 3, 1926).**

The product is prepared by mixing 52 parts, by weight, of tall oil and 50.6 parts of benzoyl chloride, condensing, then sulphonating with 83 parts of chlorosulphonic acid at a temperature of about 35°C. The product, after standing overnight, is dissolved in water and neutralized with sodium hydroxide; upon cooling, sodium sulphate and impurities are removed by filtration.

**(5) Oranienburger chemische Fabrik A.-G. The preparation of halogen-substituted organic sulfonic acids. German patent 595,800 (July 19, 1928).**

The acids are prepared by treating; at 33–35°C. with 130 parts of chlorosulphonic acid; 40 parts by weight of tall oil, 10 parts of mineral oil distillate, with an Engler viscosity of 7.5 at 20°C. and an iodine number of 5.9, and 0.5 part of manganese dioxide. After standing overnight, the reaction product is diluted with water, neutralized with lime water, and the organic salts which separate are filtered. The filtrate is evaporated to dryness under vacuum.

**(6) Oranienburger chemische Fabrik A.-G. Preparation of high molecular sulfonic acids or their salts. German patent 623,108 (April 26, 1929); (Dec. 13, 1935); C. A. 30: 2288.**

Examples are given. In one example the tall oil is mixed with lactic acid and chlorobenzene and condensed and sulphonated with chlorosulphonic acid at a temperature of about 30°C. After standing overnight, the sulphonic acid is taken up in water and purified with the calcium salt. It is transformed to the sodium salt with sodium carbonate. The solution is evaporated to dryness in a vacuum to give a powder having good stability.

**(7) Oranienburger chemische Fabrik A.-G. The preparation of condensed organic sulfonic acids. German patent 649,323 (Sept. 17, 1933).**

In the preparation of condensed organic sulphonic acids, 50 parts, by weight, of tall oil and 12.5 parts of monochloroacetic acid are condensed and sulphonated with 55 parts of pyrosulphuryl chloride at a temperature of about 40°C. After standing overnight, the product is taken up in water and neutralized with concentrated sodium hydroxide; and, after cooling, crystallized Glauber salt and impurities are removed by filtration. The sulphonate may be used either as a solution or evaporated to dryness.

**(8) Oranienburger chemische Fabrik A.-G. The preparation of surface-active sulfonation products. German patent 666,828 (April 2, 1927).**

A mixture is made of 100 parts, by weight, of liquid rosin and 150 parts of acetyl chloride, and heat is removed while mixing. This mixture is sulphonated with 100 parts of chlorosulphuric acid at 30°C., and the sulphonic acid is taken up in water and transformed, with cooling, into the ammonium or sodium salt. The sulphonate can be used as a substitute for Turkey red oil.

**(9) Getty, Edmund. The use of sulfonated tall oil in the manufacture of coated paper. *Loc. cit.*, Coating Agent (1).**

**(10) Stewart, C. W. An adhesive for paper clay coatings. *Loc. cit.*, Coating Agent (2).**

**(11) Aktiengesellshaft fuer Chemiewarte. The use of the residue from oil purification. German patent 505,930 (Dec. 18, 1924), *Chem. Zentr.* 1930, II: 2344.**

The residue obtained from oil purification is treated with sulphonated tall oil in the presence of alkali.

**(12) Foulon, A. New adhesives and accessory materials in the paint and varnish industry. Farbe u. Lack 1934: 194, 206, 219–220.**

Some new products, important to the paint and varnish industry, are discussed. Among the products discussed is sulphonated tall oil.

**(13) Keghel, M. de. The utilization of tall oil. Rev. Chim. ind. 35: 170–173, 202–206 (June, July, 1926); Pulp Paper Mag. Canada 26, no. 16: 529–531, 546, 548 (April 19, 1928); C. A. 21: 1354; T. S. 85: 212.**

The composition of tall oil distillate is given. Listed are a number of uses of the distilled fatty acids, the resin fraction, and the alkali soap. Sulphonated resin acids can be used in ready-mixed, anti-rust, and anti-fouling paints. The residual pitch, which is very similar to stearin pitch, can be used in the same kinds of paint as the sulphonated resin acids.

**(14) Pollak, Arthur. Utilization of tall oil. Oil & Soap. 17: 87–89 (1940); C. A. 34: 3492; B.I.P.C. 11: 32; B.C.P.A. 1940 B: 544.**

Tall oil is used in making asphalt emulsions, as a substitute for fatty acids in softening rubber, in the soap industry, and in the petroleum and paint industry. Sulphonated tall oil can be used as a substitute for Turkey red oil.

**(15) Argruss, Meyer S., and Schindler, Hans. The breaking of crude oil emulsions. *Loc. cit.*, Breaking of Emulsions (1).**

**(16) Argruss, Meyer S., and Schindler, Hans. Process for emulsifying oils. *Loc. cit.*, Breaking of Emulsions (2).**

**(17) Friesenhahn, P. Washing, wetting, and emulsifying agent. *Loc. cit.*, Detergents (2).**

**(18) Hastings, R. Use of tall oil for the textile industry. *Loc. cit.*, Metallic Salts (4).**

**(19) Oranienburger Chemische Fabrik A.-G. Preparation of high molecular sulfonic acids or their salts. German patent 607,018 (Feb. 2, 1927); C. A. 29: 1906.**

The method of preparation consists of treating 80 parts of tall oil and 20 parts of dark viscous lubricating oil with a mixture of 15 parts of phosphorous trichloride and 70 parts of

fuming sulphuric acid, containing 40% sulphur trioxide, at a temperature of about 35°C. Condensation and sulphonation are completed, after standing for several hours, by the addition of 90 parts of chlorosulphonic acid. The sulphonic acids are diluted in about 75% of their weight of water. This solution is heated to boiling, neutralized with lime water, and filtered from the calcium sulphate; and the hot filtrate is treated with soda. After cooling, it is filtered from the calcium carbonate and evaporated to dryness.

## *Sulphonation*

**(1) Chem. Fabrik Floersheim Dr. H. Nordlinger A.-G. A substitute for Turkey red oil. *Loc. cit.*, Emulsifying Agent (4).**

**(2) Geigy, J. R., A.-G. A sulfonated derivative of tall oil. Swiss patent 156,113 (Aug. 6, 1931); C. A. 27: 1212.**

A resinous water-soluble substance, useful in the textile industry, is obtained by the treatment of a mixture of 100 parts of tall oil and 30 parts of phenol with a sulphonating agent.

**(3) I. G. Farbenindustrie A.-G. Tall oil derivatives as dispersing agents. *Loc. cit.*, Emulsifying Agent (9).**

**(4) Oranienburger Chemische Fabrik A.-G., Linder, Kurt, and Zickermann, J. The preservation of solutions and gels of organic colloids. German patent 552,091 (Feb. 12, 1927); Chem. Zentr. 1932, II: 1575.**

Tall oil, mineral oil distillate, and chlorosulphonic acid are used in the preparation of a sulphonic acid.

**(5) Vilbrandt, Frank C., Chapman, Paul E., and Crockin, Jerome M. The sulfonation of tall oil. The separation of rosin and fat acids. Ind. Eng. Chem. 33, no. 2: 197–200 (Feb., 1941); C. A. 35: 2021; B.I.P.C. 11: 249.**

An investigation was made of the purification of tall oil by an extraction process and by the use of sulphuric acid. The product was not greatly improved using the extraction process. Varying quantities of sulphuric acid were added to a gasoline solution in the acid-refining process. To separate the fatty acids from the rosin, tall oil, which had been purified by sulphuric acid treatment, was sulphonated at low temperatures. Water-soluble sulphuric acid esters of the fatty acids were formed. Investigations were made of the effect of acid concentration at 0°C. and of the effect of time and acid quantity at —13°C. Using 90% acid at 0°C. reduced the fatty acid component without appreciably reducing the rosin content; strengths of acid below 85% gave no appearance of attack on either fatty or rosin acids. In the use of increasing amounts of 95% sulphuric acid at —13°C., the fatty acids removed were accompanied by the loss of considerable quantities of rosin.

## *Sulphur Chloride Reaction Products*

**(1) Foehre, B. A drying composition for varnish. German patent 576,262 (July 3, 1931).**

Crude tall oil is dehydrated with burnt gypsum, treated with about 8% sulphur chloride, and neutralized to produce a material for weather-resisting varnishes.

**(2) Abramowitz, William L., and Beach, Ralph M. Cutting oils from sulfurization of oils and fats. *Loc. cit.*, Cutting Oils (6).**

**(3) Daimler, Karl. Resinous masses which contain sulfur and chlorine. *Loc. cit.*, Plastic Masses (7).**

## *Sulphur Reaction Product*

**(1) Melamid, M. An oil for foundry use. *Loc. cit.*, Plastic Masses (1).**

**(2) Melamid, M. The preparation of plastic masses. *Loc. cit.*, Plastic Masses (2).**

**(3) Sanit A.-G. The preparation of plastic masses. *Loc. cit.*, Plastic Masses (5).**

**(4) Davis, Gifford D., and Barth, Edwin J. Sulfurized glyceryl esters of tall oil. *Loc. cit.*, Lubricating Oil (13).**

## *Tanning Agent*

**(1) Uddeholms A.-B. A tanning agent. Swedish patent 48,502 (April 10, 1919).**

Liquid rosin, which is emulsified with sodium hydroxide or dissolved in alcohol, is used to treat the skins either during or after tanning.

**(2) I. G. Farbenindustrie A.-G. The preparation of a tanning agent. French patent 817,477 (Feb. 8, 1937); Swiss patent 198,153; British patent 489,967; (Aug. 8, 1938); C. A. 32: 2386; 33: 1173; B.C.P.A. 1939B: 185.**

A tanning agent may be prepared by the sulphonation, using concentrated sulphuric acid, of a mixture of a natural resin, such as found in tall oil, and a phenol. Electrolytes are removed from the crude product by washing with a 2–10% aqueous solution of an alkali.

## *Textile Industry*

**(1) Henk, Hans J. Use of tall oil in the preparation of textile detergents. Seifensieder-Ztg. 67: 22 (1940); C. A. 34: 1870.**

Tall oil soap is used for varied textile requirements. Some of the uses include desizing, dyeing, degumming of silk, scouring of raw wool, washing cottons, and washing white woolens. Because it is easily oxidized and the odor is increased, tall oil soap should not be used during chlorine bleaching. Tall oil soap increases the consumption of bleach by 50% in a peroxide bleach.

**(2) Jahrstorfer, Michael, and Schwarte, Georg. Proccesses for preparing a waxlike substance. *Loc. cit.*, Hydrogenation (6).**

**(3) Riebecksche Montan-Werke A.-G. A. The preparation of fatty acid-like components. *Loc. cit.*, Hydrogenation (8).**

**(4) Hastings, R. Use of tall oil for the textile industry. *Loc. cit.*, Metallic Salts (4).**

**(5) Phrix-Arbeitsgemeinschaft. A brightening agent prepared from tall oil. Belgian patent 446,800 (Sept. 30, 1942); C. A. 39: 1064.**

A small amount of resinic acids is present in the fatty acids or their esters, obtained by the fractional distillation of tall oil. These acids, 1 gram per liter, are used in solution or emulsion, in the presence of 0.1 gram per liter of an emulsifier, to brighten textiles. Turkey red oil may be used as the emulsifier.

## *Textile Oils*

**(1) Chem. Fabrik Floersheim Dr. H. Nordlinger A.-G. A substitute for Turkey red oil. *Loc. cit.*, Emulsifying Agent (4).**

**(2) Continentale A.-G. fur Chemie und Reichsverkehrsbanke G.m.b.H. The preparation of soap and textile oils from tall oil. German patent 485,944 (Nov. 8., 1929); C. A. 24: 1241.**

Dry vegetable oils, containing free fatty acid, is mixed with tall oil, heated under vacuum, then treated with superheated steam and cooled in an atmosphere of indifferent gas to prepare oils to be used for soap making and for use with textiles.

**(3) Geigy, J. R., A.-G. A sulfonated derivative of tall oil. *Loc. cit.*, Sulfonation (2).**

**(4) Keghel, M. de. The composition of tall oil and its utilization. *Loc. cit.*, Axle Grease (1).**

**(5) Kollmann, Leo. Tall oil in textile processing. *Loc. cit.*, Cotton Bucking (1).**

**(6) Melamid, M. Preparation of fatty acids. *Loc. cit.*, Hydrogenation (7).**

**(7) Obst, W. Utilization of tall oil. Seifensieder-Ztg. 60: 856 (1933).**

A description of methods of separating the fatty and rosin acids in tall oil and of decoloring tall oil soap is given. Listed are a number of the uses of tall oil of which cutting oils is one.

## *Uses*

**(1) Bergstroem, Hilding O. V. The refining of floating resin. Svensk Papperstidn. 35: 156, 161 (March 15, 1932); C. A. 26: 3124; B.I.P.C. 2: 273.**

Suggestions are given for the commercial utilization of tall oil, and the necessity of high quality products is discussed.

**(2) Hellstroem, A. The by-products of the sulfate process. Tekn. Foeren. Finland Foerh. 36: 215–216 (1916).**

The article is a discussion of the composition, properties, and the uses of liquid rosin.

**(3) Klatt, R. Liquid rosin or tall oil. Fettchem. Umschau 41: 90–94 (1934); C. A. 28: 4901; B.C.A. 1934B: 588; T. S. 99: 240.**

A list of published analyses and tabulations of the constants of two recent tall oil samples is given, and the source, purification, and uses of tall oil are discussed. Also given is a list of 48 references.

**(4) Edwards, Karl B. The production and general characteristics of liquid rosin, or tallol. Chemistry & Industry 61, no. 21: 233–235 (May 23, 1942); Soap & Sanitary Chem. 18: 55–56 (Aug., 1942); B.I.P.C. 12: 370.**

Typical analyses of good quality liquid rosin are given for: well-prepared United States product, Swedish resinous type, Swedish medium type, Finnish medium type, and Finnish low resinous high quality type. Details are given of the purification of tall oil soap, and the acidification of the oil is discussed. A comparison is made between the tall oil produced in the U. S. and the Scandinavian material. Tall oil is a raw material for cutting and soluble oils, disinfectants, cheap paints, emulsions, and plastics, and is used in soaps where odor is not an objection.

**(5) Jennings, William H. The uses of tall oil. Paper Trade J. 120, no. 19: 41–42 (May 10, 1945); B.I.P.C. 15: 392.**

At the present time tall oil is used in the manufacture of industrial soaps, as an additive to laundry soaps, in the preparation of asphalt emulsions, in the concentration of phosphate and other ores by flotation, and in the manufacture of soluble oils by sulphonation. An ideal emulsifying agent in the manufacture of casein paints is prepared by sulphonating tall oil. Some possible future applications of tall oil are discussed.

**(6) McKee, Ralph H. The industrial uses of tall oil and its marketing problems. Southern Pulp Paper J. 7, no. 8: 14–15 (Jan., 1945); Paper Trade J. 120, no. 4: 49–50 (Jan. 25, 1945); B.I.P.C. 15: 221; C. A. 39: 1050.**

The average composition of tall oil is given; the factors influencing the future peace-time market are pointed out. Some of the demands of tall oil are in the following industries: asphalt emulsions, cutting oil, insect spray and disinfectants, lubrication grease, paint, soap, soluble oil, synthetic resins, and textile oil.

**(7) Burch, G. N. Blair, Shaw, Allan C., and Nicholls, R. V. V. The composition and potential uses of Canadian tall oils. Pulp Paper Mag. Canada 48, no. 3: 127–132 (Feb., 1947); B.I.P.C. 17: 525; C. A. 41: 3617; B. A. 1947B, II: 193.**

The potential world production of tall oil products, calculated from the production of sulfate and soda pulp, is given in tons of products. These products include crude tall oil, fatty acids, resin acids, and sterols. The tall oil recovered from black liquor is about one third of the total fatty and resin acids in freshly cut pinewood. About two thirds are lost during seasoning, pulping, and in the black liquor. Chemical constants are given for six samples of Canadian tall oils, and an analysis is reported for a commercial tall oil. Canadian oils are about three times as rich in sterols as Scandinavian oils. B-Sitosterol which is the principal phytosterol in the neutral fraction of tall oil, finds use as a substitute for cholesterol, ergosterol, or stigmasterol for the synthesis of hormones. Potential uses of the fatty and resin acids are discussed.

**(8) Byman, Leonard. Tall oil from the consumer's viewpoint. Paper Trade J. 123, no. 22: 35–40 (Nov. 28, 1946); Tech. Assoc. Papers 29: 463–469 (June, 1946); B.I.P.C. 17: 204; C. A. 41: 599.**

The article is a general discussion of tall oil markets, present and possible uses, and tall oil versus competitive materials. 111 references are given.

**(9) Gaffney, Mary, and Cargill, Ruth. A review of production, exports, stocks and future outlook for prices of rosin. Soap Sanit. Chemicals 22, no. 11: 33–36, 137 (Nov., 1946); B.I.P.C. 17: 197.**

Tall oil production is about 20% of the combined gum and wood rosin production of which about one third is consumed by the soap industry. Production figures are given for refined tall oil for the years 1940–46.

**(10) Jamieson, George S. Vegetable fats and oils. New York, Reinhold Publ. Co., 1943.**

A discussion of tall oil and its range of characteristics are given. Tall oil may be used in the preparation of asphalt, tar, cutting oils, textile soaps, driers, sulphonated oil, and drying oil. The pitch obtained in the distillation of tall oil may be used in the manufacture of ink.

**(11) Lassenius, T. The by-products of the cellulose industry. Finnish Paper and Timber J. 26, no. 6: 73–77; no. 7: 85–87 (March 31, April 15, 1944); B.I.P.C. 15: 189; C. A. 39: 616.**

Some of the uses of tall oil are in dyestuffs, leather, rubber industries, soap, and as a sizing for paper. The purification of tall oil is briefly discussed.

**(12) Annand, R. L. Liquid rosin—tall oil. Oil Colour Trades J. 112, 1319–1320, 1322 (1947); C. A. 42: 1746.**

The article is a lecture which includes a brief description of the Kraft process, the characteristics of liquid rosin, and uses of liquid rosin and its derivatives in the paint and varnish industry.

**(13) Jahn, E. C. Chemical production from Scandinavian pulp mill by-products. Paper Mill News 71, no. 10, 14, 16, 20 (1948); C. A. 42: 3959.**

Chemicals are produced from tall oil and from ethyl alcohol obtained from fermentation of sulphite waste liquor. Other products obtained are cymene, cellulose derivatives, lignin, and yeast.

## *Varnish Remover*

**(1) Hodges, Paul. A paint, varnish, and lacquer remover. *Loc cit.*, Paint Remover (1).**

## *Waterproofing Agent*

**(1) I. G. Farbenindustrie A.-G. The preparation of waterproof products. German patent 429,875 (Sept. 25, 1924).**

In the preparation of waterproof products, a mixture of fatty and resin acids are used.

**(2) Ohlsson, K. J. T. Preparation of waterproof paperboard of abric. Swedish patent 83,410 (May 21, 1935); C. A. 31: 2822; T. S. 103,215.**

An impregnated material, which can be painted, is prepared by impregnating the material with crude or refined liquid rosin obtained as a byproduct in the manufacture of sulphate pulp.

**(3) Schmid, W. Liquid rosin and its uses. *Loc. cit.*, Flypaper (1).**

## *Wetting Agent*

**(1) Boehme A.-G., H. Th. Capillary active ester-like sulfuric acid compounds. *Loc. cit.*, Emulsifying Agent (3).**

**(2) Edwards, Karl B. The manufacture of wetting agents. British patent 538,581 (July 29, 1940); B.C.P.A. 1941B, II: 384.**

A brown precipitate is obtained from waste aqueous liquor, obtained in the sulphate process, by removing the tall oil soaps, clarifying, then neutralizing with a mineral acid or acid-reacting salt of mineral acid. A wetting and foaming agent is obtained on separation and solution in aqueous alkali. The precipitate is purified by solvent extraction, solution in alkali, and reprecipitation.

**(3) Friesenhahn, P. Washing, wetting, and emulsifying agent. *Loc. cit.*, Detergents (2).**

**(4) Johnson, James M. Use of amine soaps as surface-active agents. *Loc. cit.*, Bitumen (1).**

**(5) Johnson, James M. A bituminous composition which has increased adhesion to mineral aggregate. U. S. patent 2,426,220 (Aug. 26, 1947); B.I.P.C. 18: 139; C. A. 41: 7700.**

Bituminous products, which contain the reaction product of certain amines with an acid, find use as a coating agent or bonding material with all types of wet aggregates. The amine is prepared by warming a higher fatty acid, such as tall oil or red oil, with an equimolecular proportion of an alkylene polyamine. For example; 0.5 part of a mixture of 16 parts of Napco C.V.T. and 84 parts of tall oil is used with 100 parts of asphalt.

## *Wool Scouring*

**(1) Kollmann, Leo. Tall oil in textile processing. *Loc. cit.*, Cotton Bucking (1).**

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## Calendar of TAPPI Meetings

### NATIONAL

Annual Meeting: Commodore Hotel, New York, N. Y. February 20–23, 1950.

Fundamental Research Conference: Chateau Frontenac, Quebec, P. Q., May 29–30, 1950. Symposium: "The Papermaking Properties of Fibers." (Joint Meeting with Technical Section, Canadian Pulp and Paper Association).

Fifth Engineering Conference of the Pulp and Paper Industry. Netherland Plaza Hotel, Cincinnati, Ohio, October 2–5, 1950.

### LOCAL SECTIONS

PACIFIC SECTION:

April 7, 1950, Camas, Wash.

EMPIRE STATE SECTION:

*Western New York Group:*

March 8, 1950, Prospect House, Niagara Falls, N. Y.
April 12, 1950, Spaulding Fibre Co., Tonawanda, N. Y.
May 10, 1950, Prospect House, Niagara Falls, N. Y.

*Metropolitan Group:*

March 14, 1950, Fraunces Tavern, New York, N. Y.

*Northern New York Group:*

March 9, 1950, Woodruff Hotel, Watertown, N. Y.
April 13, 1950, Woodruff Hotel, Watertown, N. Y.

DELAWARE VALLEY SECTION:

March 23, 1950, Engineers Club, Philadelphia, Pa.
April 27, 1950, Engineers Club, Philadelphia, Pa.

OHIO SECTION:

March 14, 1950, Manchester Hotel, Middletown, Ohio.
April 13, 1950, Manchester Hotel, Middletown, Ohio.
May 11, 1950, Manchester Hotel, Middletown, Ohio.

MAINE-NEW HAMPSHIRE SECTION:

June 23–24, 1950, Wentworth-by-the-Sea, Portsmouth, N. H.

KALAMAZOO VALLEY SECTION:

March 2, 1950, Columbia Hotel, Kalamazoo, Mich.
April 6, 1950, Harris Hotel, Kalamazoo, Mich.
May 4, 1950, Harris Hotel, Kalamazoo, Mich.

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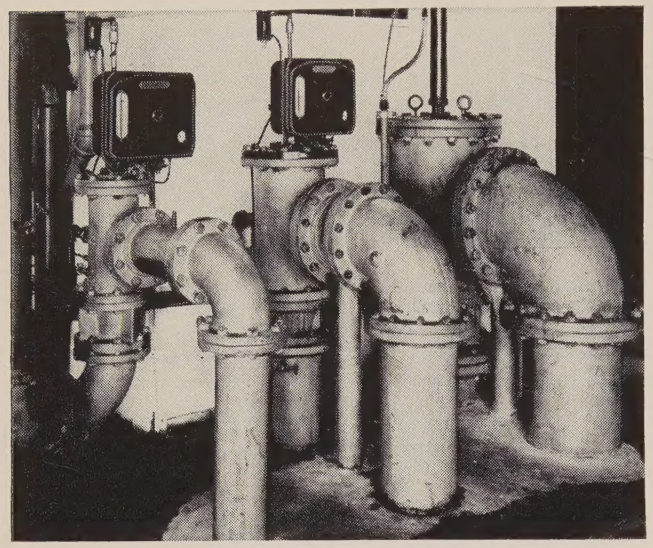

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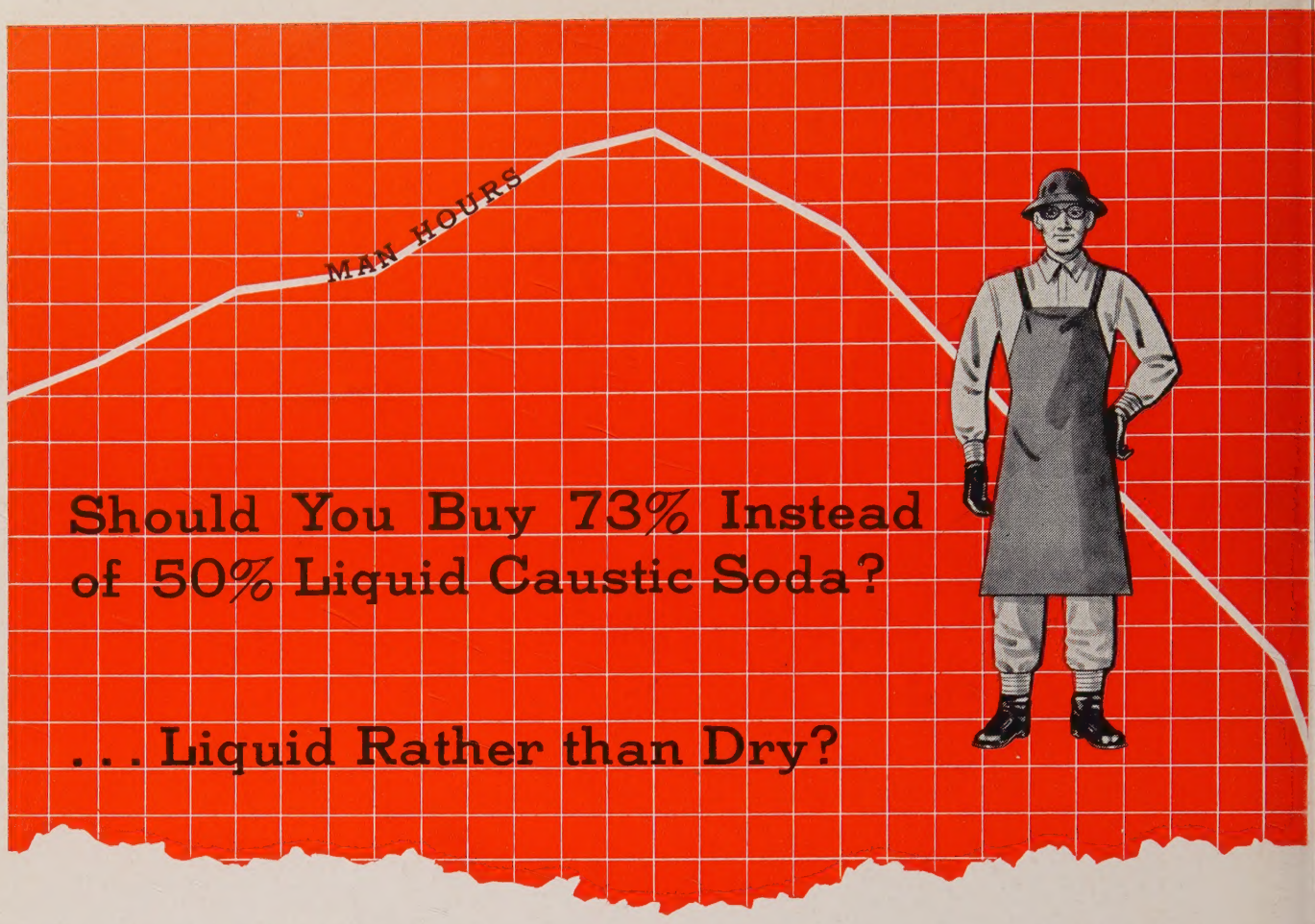


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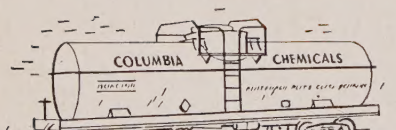


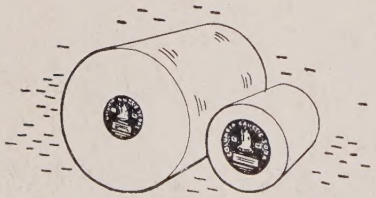

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